

**Final Technical Report**  
**for**  
**A Pilot-Scale Evaluation of Polychlorinated Biphenyl Bioremediation**  
**Technologies**  
**at**  
**Cape Canaveral Air Station, Florida**



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September 28, 2000

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Dear Mr. Hansen: .

Attached please find one copy of the Final Technical Report for a Pilot-Scale Evaluation of Polychlorinated Biphenyl Bioremediation Technologies at Cape Canaveral Air Station, Florida. The report has been revised to include your comments and those received from Don Kampbell. If you have any questions or comments, please feel free to call me at 614-424-7771.

It was a pleasure working with you on this effort.

Sincerely,

A handwritten signature in dark ink, appearing to read "Jeff J. Morse", with a long, sweeping horizontal line extending to the right.

Jeff J. Morse  
Research Scientist  
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Enclosures (1)

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## ABBREVIATIONS AND ACRONYMS

HDPE	high-density polyethylene
GC/MS	gas chromatography/mass spectroscopy
ND	not detected
NM	not measured
PCB	polychlorinated biphenyls
ppm	parts per million
PVC	polyvinyl chloride
RSKSOP	R.S. Kerr Standard Operating Procedure
$t_0$	time zero
U.S. EPA	United States Environmental Protection Agency

## 1.0 INTRODUCTION

Polychlorinated biphenyls (PCBs) are a group of chemicals possessing chemical properties that make them attractive for use in a wide variety of industrial applications. Their widespread use has led to their introduction into the environment. The Air Force has a number of sites that are contaminated with PCBs. This contamination has typically resulted from leaks and spills of electrical transformer cooling oils that contained PCBs. The potential harm posed to human health and the environment has prompted the United States Environmental Protection Agency (U.S. EPA) and state regulatory authorities to set maximum allowable PCB concentrations at a site.

In the past, remediation of PCB-contaminated sites typically consisted of excavating the contaminated soil and either incinerating it or shipping it to a hazardous waste landfill for disposal. The expense involved with this process coupled with existing evidence that PCBs may be biologically degraded has led the Air Force to investigate biodegradation as an alternative to conventional remediation strategies.

Previous research has demonstrated the potential to biologically degrade PCBs under both laboratory and field conditions, but the success of bioremediation applications depends heavily upon site-specific conditions, so it is advisable to perform treatability testing when considering it as a treatment alternative. This report details the design, operation and results of an 18-month biological treatability test conducted on PCB-contaminated soil at Cape Canaveral Air Station, FL.

The objective of this study was to evaluate the potential of three bioremediation techniques for use in treating soils contaminated with PCBs at Air Force sites. The three techniques tested included natural attenuation, sequenced anaerobic and aerobic treatment, and addition of a commercially available microbial amendment product. The success of each technique was based on the reduction of PCB contamination in soil over the duration of the test.

## **2.0 TECHNICAL APPROACH**

PCB-contaminated soils excavated from a contaminated launch complex at Cape Canaveral Air Station underwent field testing to assess the potential effectiveness of three biologically based PCB treatment technologies. The three technologies were tested in duplicate in six aboveground test cells, each containing approximately 9 ft<sup>3</sup> of the PCB-contaminated soil. Testing was performed on site at Cape Canaveral's Fire Training Area 17 over a period of approximately 18 months. Testing conditions and treatment effectiveness were monitored through both field measurements and analyses of soil samples collected during the experiment. Testing under field conditions provided an assessment not only of each technology's effectiveness, but also its practicality.

### **2.1 The Treatment Conditions**

The three biological treatment technologies selected for testing included natural attenuation, sequenced anaerobic and aerobic treatment, and use of the commercially available microbial amendment product X-19. All three treatments were set up in parallel.

The first treatment condition was a no-treatment control used to determine the background rate of PCB biodegradation in the contaminated soils. Although the soils in this condition had been disturbed during excavation and subsequent homogenization, it was used as an indicator for the potential for natural attenuation of the PCBs.

The second treatment condition examined the effectiveness of the commercially available microbial amendment X-19, which had shown some promise for PCB remediation of soils in the laboratory. The amendment was used in strict accordance with manufacturer's specifications. The results from this condition demonstrated the effect of adding exogenous microorganisms and other amendment components on the rate and extent of PCB biodegradation.

The third treatment condition consisted of a sequential anaerobic/aerobic process. The anaerobic phase was designed to reductively dechlorinate the more heavily chlorinated PCB congeners. The electron-donating substrates potassium acetate and sodium butyrate were added to the sequential test cells during this phase to deplete the supply of electron acceptors in the soil and promote reductive dechlorination.

In the second part of the sequential process, the test cells were opened and aerated. This aerobic incubation provided the opportunity for aerobic microorganisms to degrade the remaining less-chlorinated congeners. In a final effort to stimulate aerobic biodegradation, biphenyl and nutrients were added to the sequential test cells. Results from the sequential testing condition provided information about the ability of indigenous microorganisms to degrade PCBs under aerobic and anaerobic conditions and when provided various substrates and nutrients.

### **2.2 Analytes**

The experimental design for this study included a monitoring strategy aimed at tracking not only changes in total PCB mass, but also changes in the PCB fingerprint and selected biologically important parameters. Test cells were routinely analyzed using both field and laboratory based analyses to provide information about conditions within the cells and the effectiveness of the treatment. Field measurements of soil-gas oxygen, moisture content and temperature were taken in all six cells regularly. In addition, soil samples collected from the cells were sent to the U.S. EPA's R.S. Kerr Laboratory in Ada, Oklahoma, to undergo a suite of laboratory-based analyses. The analytical regiment for each testing condition is outlined in Table 1 below.

**Table 1. Analytical Regiment Conducted on each Treatment Condition**

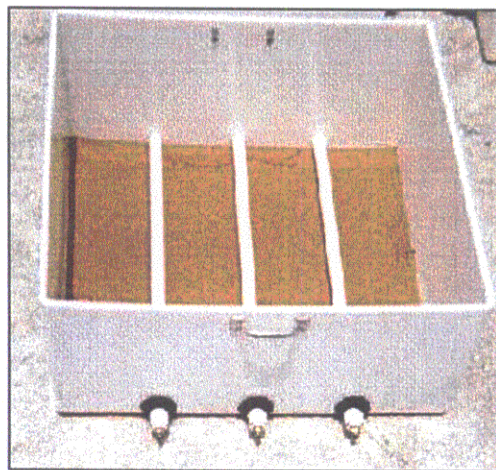
	<b>Control/Natural Attenuation Cells</b>	<b>X-19 Treated Cells</b>	<b>Anaerobic/Aerobic Cells</b>	
<b>Laboratory-Based Measurements</b>	PCB Congeners	PCB Congeners	PCB Congeners	Acetic acid
	Soil moisture	Soil moisture	Soil moisture	Butyric acid
	Soil pH	Soil pH	Soil pH	Nitrate
	Chloride	Chloride	Chloride	Sulfate
<b>Field Monitoring Parameters</b>	Soil-gas oxygen Soil moisture Temperature			

### 3.0 METHODS

#### 3.1 Test Cell Design, Construction and Installation

Six test cells were constructed at the Battelle Memorial Institute in Columbus, Ohio. Each cell was 3 ft wide, 3 ft long, and 16 inches deep. Cells were constructed of high-density polyethylene (HDPE) and the bottom of each cell was lined with a ¼-inch thick cork sheet. The cork served to prevent the loss of soil during sampling events by forming a plug in the bottom of the coring device. In addition, four of the six cells had three 1-inch-diameter, 33-inch-long, 0.010-slot polyvinyl chloride (PVC) screens inserted through one side of the cell to allow for aeration (see Figure 1). Lids were constructed for the sequential test cells so anaerobic conditions could be maintained during the anaerobic phase of that treatment condition.

Approximately 2½ yd<sup>3</sup> of PCB-contaminated soil was collected from soil stockpiles slated for treatment at the former soil washing operation located at Fire Training Area 17. To reduce PCB concentration variability, the soil was mixed as thoroughly as possible with a front-end loader and shovels before adding any amendments and/or loading it into the treatment cells. This homogenized soil served as the source of material for each of the treatment cells.



**Figure 1. Photograph of Test Cell before Filling with PCB-Contaminated Soil**

The test cells were designed to prevent the release of PCB-contaminated soils or water and consequently lacked a mechanism to naturally drain excess water. As a result, a water-resistant tarp was used to cover the test cells after setup and prevent rain from accumulating within the cells.

##### 3.1.1 Setup of Control/Natural Attenuation Test Cells

The two control/natural attenuation test cells were prepared on 14 July 1998 by adding approximately 9 ft<sup>3</sup> of the homogenized PCB-contaminated soil to the two treatment cells that did not contain PVC ventilation screens. Both cells had three oxygen sensors installed at depths of 3, 6, and 9 inches during soil addition. Approximately 10-L of water were added to each of the two cells to raise their moisture content to approximately 60% of field capacity. No other amendments were added.

##### 3.1.2 Setup of X-19 Amended Test Cells

The primary concern in setting up the X-19 amended test cells was following the vendor's instructions to ensure the product was tested fairly. The following vendor-provided instructions were used for preparing the X-19 test cells:

1. Add one part X-19 to two parts soil by volume, to achieve a mixture that is approximately 30% X-19 by volume.
2. Once the soils have been sufficiently mixed, raise the moisture content of the mixture to approximately 30 percent field capacity using an aqueous solution containing 100 parts per

million (ppm) ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ) and 100 ppm potassium dihydrogenphosphate ( $\text{KH}_2\text{PO}_4$ ).

3. Cover the soil/X-19 mixture with an impermeable plastic layer such as Visqueen™ to prevent moisture loss.
4. Do not aerate the cells.

Complying with these instructions required mixing the homogenized PCB-contaminated soil with the X-19 at the correct proportions. This was accomplished by adding one 5-gallon bucket full of X-19 for every two 5-gallon buckets full of PCB-contaminated soil to the bucket of a clean front-end loader. The X-19 and soils were then mixed using shovels, rakes and hoes. Because the contaminated soil was lighter in color than the X-19 it was easy to see unmixed portions of soil. The mixture was agitated until it was a uniform color.

After the contaminated soil and X-19 had been effectively homogenized, two 10-L batches of aqueous solution containing 100 ppm ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ) and 100 ppm potassium dihydrogenphosphate ( $\text{KH}_2\text{PO}_4$ ) were prepared. The solution was sprayed onto the soil/X-19 mixture with an aerosol garden sprayer while the mixture was being turned with shovels. Once the solution had been applied, the mixture was sufficiently moist and well mixed to be added to the X-19 test cells. Approximately  $9 \text{ ft}^3$  of the mixture and three oxygen sensors at depths of 3, 6, and 9 inches were added to each X-19 test cell. Finally, a thin sheet of plastic was placed over the soil/X-19 mixture and secured with rocks (see Figure 2). Since the vendor requested that the cells not receive aeration, the aeration screens were sealed throughout test.



**Figure 2. Photograph of X-19 Amended Cell after Setup**

### **3.1.3 Setup of Sequential Test Cells**

The sequential test cells were the last cells to be setup, but the remaining supply of homogenized PCB-contaminated soil was not sufficient to fill the cells to the desired level. Consequently, additional contaminated soil was obtained from the original stockpile location and homogenized as before. After the contaminated soil had been homogenized, two 10-L aqueous solutions containing 100 mM potassium acetate and 100 mM sodium butyrate were prepared. Ten liters of this solution was sprayed onto the soils in each cell as they were being loaded. In addition, three oxygen sensors were installed in each cell at depths of 3, 6, and 9 inches, respectively. After the sensors were in place, a tight-fitting lid was lowered into place and the aeration screens sealed with  $\frac{1}{2}$ -inch pipe plugs. These precautions were intended to prevent oxygen from diffusing into the cells during the anaerobic phase of the experiment.

## **3.2 Test Cell Operation**

Field testing began on 15 July 1998 with the collection of the first round of soil samples from each of the six newly installed test cells (see Figure 3). The field test was initially designed to run for six months with a minimum of on-site supervision. Four site visits were planned, during which test cells were sampled and monitored for soil-gas oxygen concentration, temperature, and soil moisture. In addition, maintenance activities such as the addition of nutrients or the adjustment of soil moisture content were also performed.



**Figure 3. Photograph of Test Cells**

Because the cells were covered to minimize the potential for saturation by rainfall, cells tended to slowly lose soil moisture over the course of the experiment. To compensate for this phenomenon, water was occasionally added to restore the moisture level to its initial level. On two occasions, particularly violent storms tore portions of the protective tarp from its foundations and allowed heavy rains to inundate some of the cells with water. These cells were drained by inserting a small section of well screen into each corner of the saturated cell and pumping out the excess water with a peristaltic pump. Alternatively, cells were drained through the existing PVC screens that were installed during cell construction to provide aeration.

When results from the third round of soil samples showed no significant changes in the PCB profiles from any of the treatment conditions, the length and scope of the experiment was extended. Eventually eight rounds of samples were collected over an 18-month period. The final round of samples was collected on 18 January 2000.

### **3.2.1 Operation of Control/Natural Attenuation Cells**

The installation of the control/natural attenuation test cells N1 and N2 was completed on 14 July 1998. Both cells operated with minimal human intervention until Cell N1 was found inundated with water on 2 November 1999 (Day 475). The cell was drained that day and operation continued as before. The only other significant deviation from the planned treatment was its extended duration. The cells were disassembled on 18 January 2000 (Day 552).

### **3.2.2 Operation of X-19 Cells**

The setup of the X-19 amended cells was finished on 14 July 1998 as described in Section 3.1.2. As directed by the vendor, cells were operated without aeration and no additional nutrients were added after setup. Although every attempt was made to operate the cells to vendor specifications, heavy rains flooded both cells on 9 November 1998 (Day 117). The cells were drained on 19 November 1998 (Day 127). On

1 February 1999 (Day 201) both X-19 treated cells were mixed using shovels, hoes and rakes. Cell X2 was found flooded again on 2 November 1999 (Day 475). It was drained the same day. The cells were disassembled on 18 January 2000 (Day 552).

### **3.2.3 Operation of Sequential Cells**

The installation of Cells A1 and A2 was completed on 15 July 1998 as described in Section 3.1.3. The cells operated for 202 days under anaerobic incubation, followed by 274 days of aerobic incubation and finally 76 days of nutrient amended aerobic incubation. The operational details for each of these treatments are provided below.

The sequential cells operated in anaerobic mode for 202 days, until 2 February 1999. The cells were opened twice during the anaerobic incubation for feeding. Each cell was fed a 3-L aqueous solution containing 100g potassium acetate and 100g of sodium butyrate on 14 October 1998 (Day 91) and again on 9 November 1998 (Day 117). The solution was sprayed onto the soils with an aerosol garden sprayer while they were being mixed with shovels and rakes. This application method temporarily returned the cells to an aerobic state, but was necessary to ensure the uniform distribution of electron donors.

On 2 February 1999, the sequential test cells were converted from anaerobic treatment to aerobic treatment. The lids were removed and a blower was connected through a manifold system to the aeration screens in the bottom of each cell. Airflow to the cells was maintained at 3-5 ft<sup>3</sup>/hour by six rotameters on the manifold. The blower operated intermittently due to mechanical difficulties until it was disconnected after 72 days of operation on 15 April 1999.

Cell A1 was found with ponded water covering about 1/3 its surface on 2 November 1999 and was drained the same day and prepared for a third and final treatment technique.

On 3 November 1999 both sequential cells were amended with biphenyl and nutrients to see if biological PCB-degrading activity could be stimulated. Each cell received a 1-L aqueous solution containing 43g of NH<sub>4</sub>NO<sub>3</sub> and 7g of (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> and a 1-L solution consisting of 89g of biphenyl dissolved in acetone. Furthermore, the addition of acetate and butyrate during the anaerobic phase of the experiment caused the pH in the sequential treatment cells to rise above desired levels; consequently, a pH adjustment was made by adding an 8-L aqueous solution containing 101g of concentrated HCl to each cell. Aeration was reinitiated on 4 November 1999 (Day 477) at the more conservative flowrate of approximately 5 ft<sup>3</sup>/day. Compressed air tanks were used to provide a more reliable source of ventilation.

The sequential cells were disassembled after 552 days of operation on 18 January 2000.

### **3.3 Soil Sampling**

Soil samples were collected with a hand-driven stainless steel core barrel fitted with a 12-inch-long, 1-inch-diameter plastic sleeve liner. After sampling, the sleeve was removed from the core barrel and sealed with polyethylene caps and tape. The sample was labeled and all pertinent information recorded in the field logbook. Sealed cores were immediately put into a cooler with Blue Ice™ and shipped via overnight delivery to the U.S. EPA's R.S. Kerr Laboratory for analysis.

Initially seven soil cores were collected from each test cell, but when results from the first two sampling events showed reasonably tight confidence intervals, the number of cores collected was reduced to five.

### 3.4 Analytical Methods

Experimental data were collected using both field- and laboratory-based methods. The following subsections describe the methods used to monitor test cell parameters in the field and obtain laboratory-based analytical data.

#### 3.4.1 Field-Based Monitoring Methods

Three parameters were measured in the field: soil-gas oxygen, temperature, and soil moisture content. Soil-gas oxygen was monitored using Figaro Model KE-25 oxygen sensors. Three sensors were installed in each cell during test setup at depths of 3, 6 and 9 inches. The sensors provided a millivolt response that was read by connecting a voltmeter to leads from each probe. Each probe's millivolt response was recorded in the field notebook and the oxygen concentration was extrapolated from the calibration curve for each sensor.

Soil temperatures were measured by inserting a temperature probe to a depth of 4 inches approximately midway between the center of each cell and one of the cell walls. The final temperature reading was recorded in the field notebook after the digital probe readout had stabilized.

Soil moisture was assessed in the field using a soil moisture probe. The probe was inserted to a depth of about 6 inches approximately midway between the center of each cell and one of the cell walls. The probe was used in accordance with the manufacturer's specifications. The digital probe readout was allowed to stabilize before the final value was recorded in the field notebook.

#### 3.4.2 Laboratory-Based Analytical Methods

Table 1 outlines the analytical methods, sample volume requirements, container types, preservation methods and holding times that were used in this study. Appendix A contains the referenced R.S. Kerr Standard Operating Procedure (RSKSOP-177) and Dionex Application Note 316.

**PCB Congener Analysis.** Evaluating the effectiveness of each treatment required analyzing for specific PCB congeners over the treatment period. Soil samples were extracted using the method described in the "Dionex Application Note 316 Extraction" and then analyzed by gas chromatography/mass spectroscopy (GC/MS) following SW-846 Method 8082.

**Acetate and Butyrate.** The electron donors acetate and butyrate were quantified using RSKSOP-177, *Quantitation of Phenols and Aliphatic and Aromatic Acids as Pentafluorobenzyl Ethers or Esters by Negative Ion Chemical Ionization Gas Chromatography/Mass Spectroscopy*. A copy of this method is can be found in Appendix A.

**Nitrate Analysis.** The electron acceptors nitrate was quantified in soil samples taken from the sequential treatment cells during the anaerobic incubation. The Lachat colorimetric-hydrazine reduction method was used.

**Chloride and Sulfate Analysis.** The determination of chloride and sulfate concentrations in soil samples required samples to be extracted with ultra-pure water. The extract was then analyzed with capillary electrophoresis.

**Table 2. Summary of Laboratory Analyses, Sample Volume Requirements, Container Types, Preservation Methods, and Holding Times**

Parameter	Method	Sample Volume	Container	Holding Time/ Preservation
PCB Congeners	Extraction: Dionex Application Note 316 Extraction	100 g	Glass	7 days at 4°C
	Analysis: EPA Method 8082			
Cl, SO <sub>4</sub>	1:1 Extraction with Water	10 g	Plastic or glass	14 days
	Analysis: Waters Capillary Electrophoresis			
Acetate, Butyrate	RSKSOP-177	50 g	Glass	24 hours
NO <sub>3</sub>	Lachat colorimetric-hydrazine reduction	10 g	Plastic or glass	24 hours at 4°C
Soil Moisture	Gravimetric: dry at 105°C	10 g	Plastic or glass	24 hours
Soil pH	pH probe in 1:1 aqueous paste	10 g	Plastic or glass	24 hours at 4°C

**Soil Moisture Analysis.** A gravimetric method was used to determine the moisture content of soil samples. Approximately 10 g of sample was weighed upon arrival at the laboratory. The sample was weighed again after drying at 105°C for at least 4 hours and then cooling to ambient temperature in a dessicator. The cycle of drying and dessicating was repeated until the difference between successive measurements differed by less than 5 mg.

## 4.0 RESULTS

### 4.1 PCBs

This section presents the results of the PCB congener analyses performed on soil cores obtained from each of the treatment conditions. The data are presented in two graphical formats. The first format shows the total of all quantified PCB congeners and is used to assess the overall mass removal of PCBs over the course of the treatability test. The second format shows the congener fingerprint of a soil sample and displays the quantity of each individual congener present in the sample. Such graphs are used to observe shifts in congener distribution. This type of examination is particularly important for anaerobic treatments, which often show only slight overall mass reduction, but do transform the more highly chlorinated congeners to less-chlorinated congeners. Such a transformation would be illustrated by a shifting of congener masses from right to left on the graph.

Soil cores were analyzed for all 209 PCB congeners, but only 35 congeners were detected and therefore reported in data tables and graphs. This group of congeners consisted exclusively of tri, tetra and pentachlorobiphenyls. The congeners are identified on fingerprint graphs by a numeric designation, in which the location of chlorine atoms is identified. For example, the trichlorobiphenyl identified as "26-4" has a chlorine atom at the two ortho positions (position 2 and 6) on one ring of the biphenyl backbone and a third chlorine atom at the para position (position 4) on the other ring. See Figure 4 below.

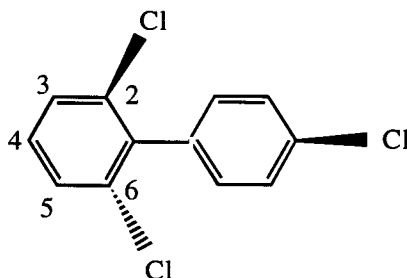
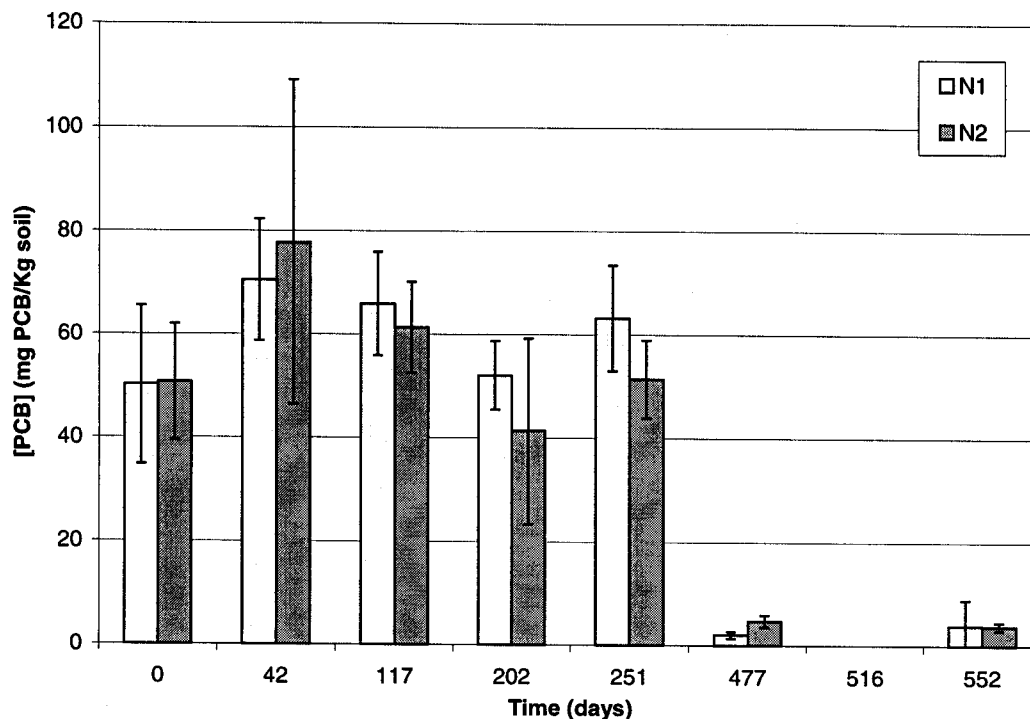


Figure 4. Illustration of Trichlorobiphenyl 26-4

Initial total average PCB concentrations in samples ranged from 26 ppm in Cell X1 to 161 ppm in Cell A2. As expected, the cells amended with X-19 had lower initial PCB concentrations resulting from the dilution of contaminated soil with the X-19 product matrix. Conversely, the sequential cells A1 and A2 had the highest initial PCB concentrations; the PCB concentration in Cell A2 was 2 to 3 times greater than any other treatment cell. In general, the reproducibility between duplicate cells was excellent. Specific results from each of the three treatment conditions are provided in the following subsections.

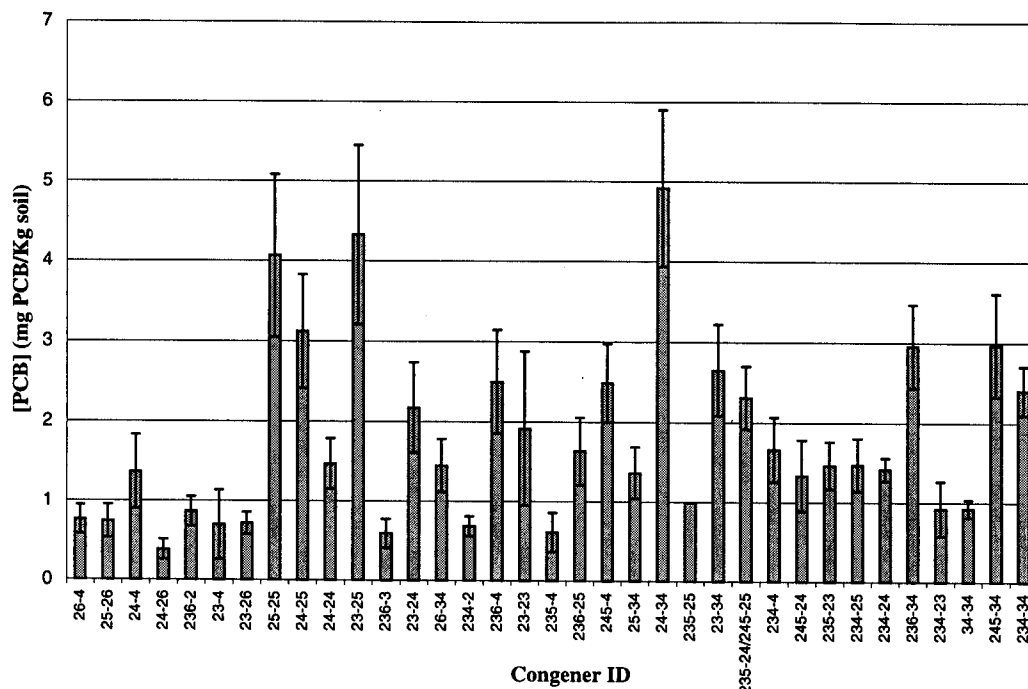
#### 4.1.1 Control/Natural Attenuation Cells

The total average PCB concentration in Cells N1 and N2 remained fairly constant over the first 251 days of the experiment, with values ranging between 40 and 80 ppm; however, a dramatic decrease (> 90%) was observed in subsequent sampling events (see Figure 5). The reason for this sudden change is unknown, but the fact that a similar change occurred in all three treatment conditions simultaneously suggests that the change is a result of an analytical inconsistency (e.g., internal standard recovery correction, dilution factor, extraction efficiency, etc.) The results from the duplicate cells N1 and N2 were very similar.

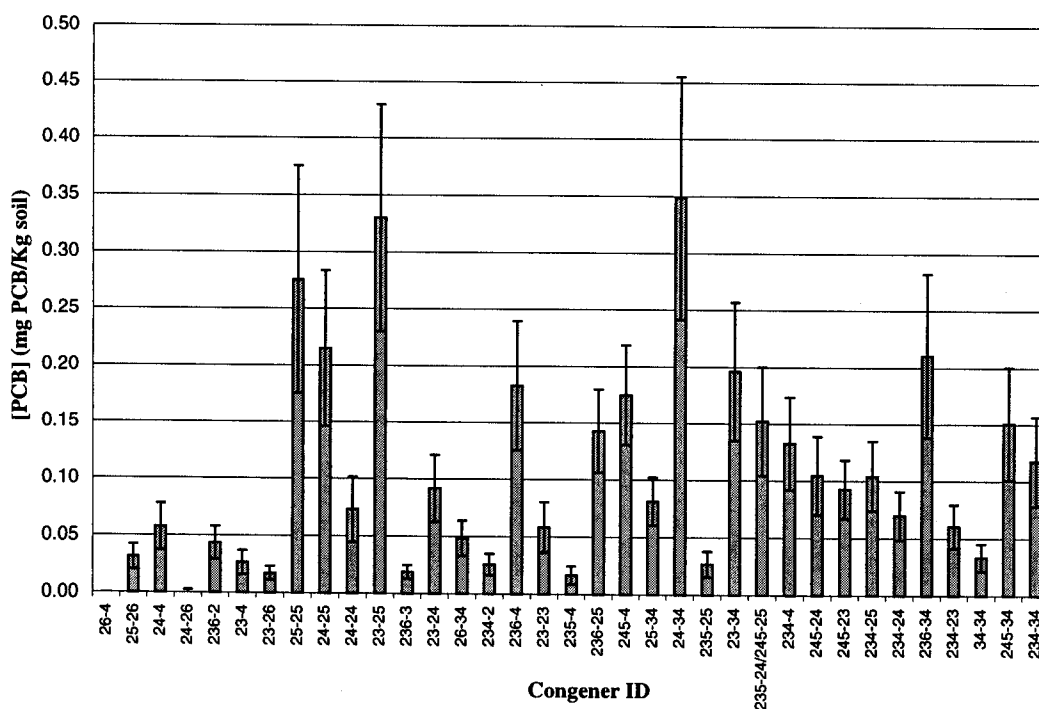


**Figure 5. Total Average PCB Concentration in Control/Natural Attenuation Test Cells**  
(Error bars represent 95% confidence intervals [n = 5-7] Data from the Seventh Sampling Event [Day 516] was unavailable at the time this report was written.)

Significant changes in the PCB congener fingerprint of the control test cells were not expected and did not occur. With very few exceptions, the relative distribution of one congener to the next remained nearly constant. In addition, individual congener concentrations changed proportionally with the total average PCB concentration. These findings reinforce the notion that PCBs are particularly recalcitrant environmental contaminants. Figures 6 and 7 show the initial and final congener fingerprints from Cell N2, respectively.



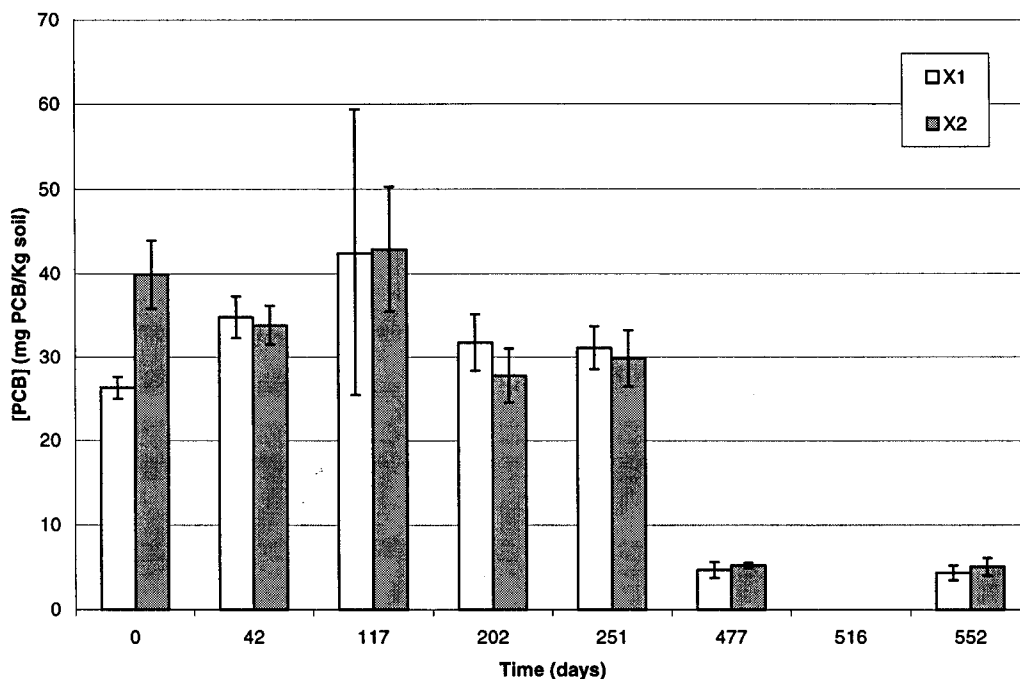
**Figure 6. Time Zero ( $t_0$ ) Congener Fingerprint for Cell N2**  
(Error bars represent 95% confidence intervals [n=7])



**Figure 7. Final ( $t_7$ ) Congener Fingerprint for Cell N2**  
(Error bars represent 95% confidence intervals [n=4])

#### 4.1.2 X-19 Treatment

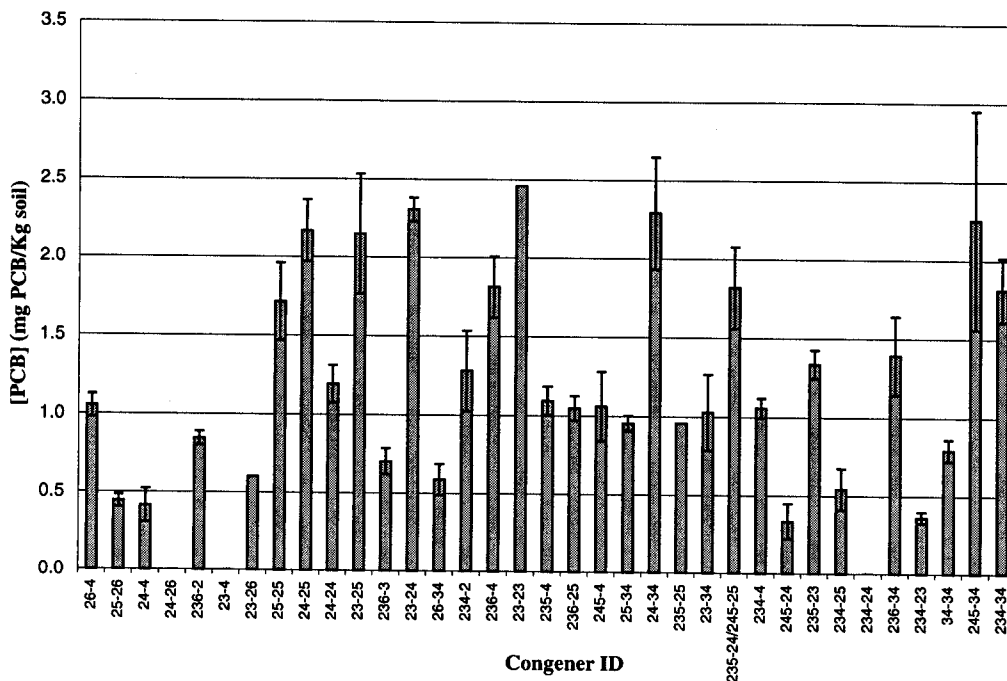
The total average PCB concentration in Cells X1 and X2 ranged between 26 and 43 ppm over the first 251 days of the experiment. Although the average PCB concentration did fluctuate during that time, no obvious reduction in the overall PCB concentration was apparent. As with the other treatments, a significant decrease in the PCB concentration was observed after the fifth sampling event. Cells X1 and X2 demonstrated an 85% and an 82% reduction in total average PCB concentration respectively. The overall results from the duplicate cells (X1 and X2) were very similar.



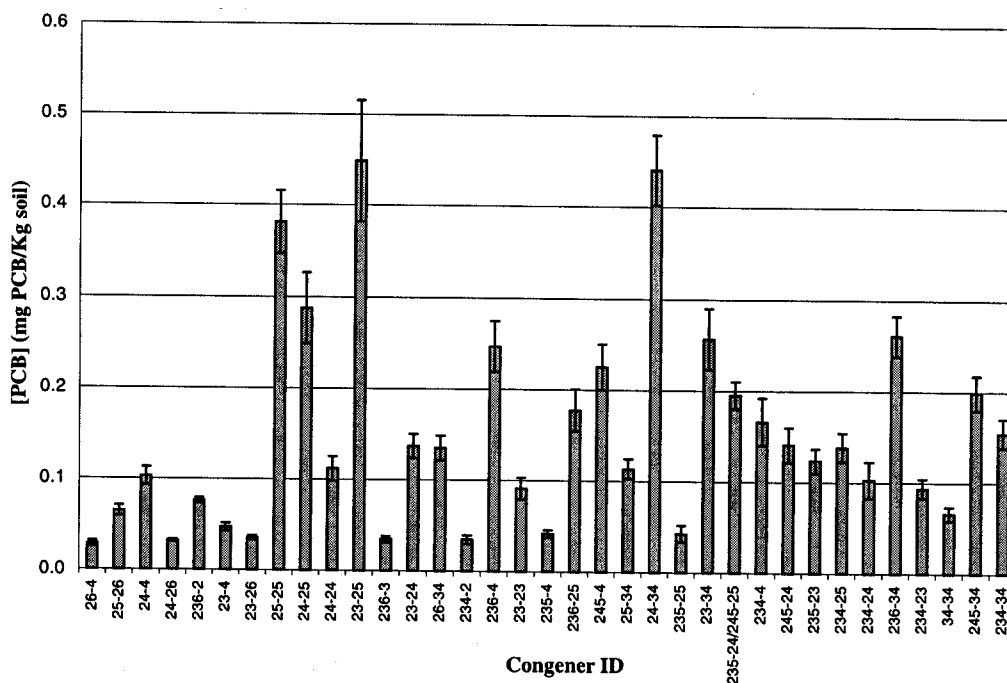
**Figure 8. Total Average PCB Concentration in X-19 Treated Cells**  
(Error Bars represent 95% confidence intervals [n = 3-7]. Data from the Seventh Sampling Event [Day 516] was unavailable at the time this report was written.)

Surprisingly, the initial PCB congener fingerprint for Cells X1 and X2 did not fit the profile observed in the other treatment conditions. This unusual finding was short lived, however, as all subsequent sampling events showed the distinctive congener profile for this contaminated soil. The reason for this anomaly remains unknown, but may be linked to the partitioning of the PCB congeners into the peat matrix of the X-19. Differential rates of sorption may have affected the extraction efficiency of the PCB analysis and provided a skewed initial fingerprint.

With the exception of the initial sampling event, the relative proportion of individual congeners remained stable throughout the experiment. As with the other two treatment conditions, the overall mass reduction observed towards the end of the experiment demonstrated a proportional reduction in each congener, which suggests that a process other than biotransformation was responsible.



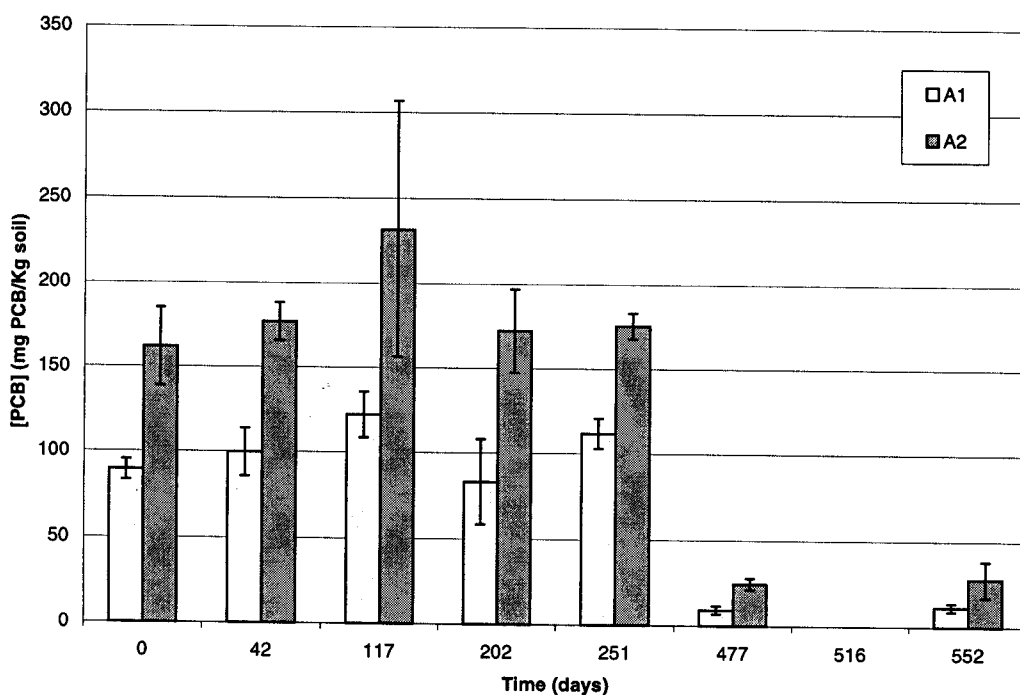
**Figure 9. Time Zero ( $t_0$ ) Congener Fingerprint for Cell X2**  
(Error bars represent 95% confidence intervals [ $n=7$ ])



**Figure 10. Final ( $t_7$ ) Congener Fingerprint for Cell X2**  
(Error bars represent 95% confidence intervals [ $n=3$ ])

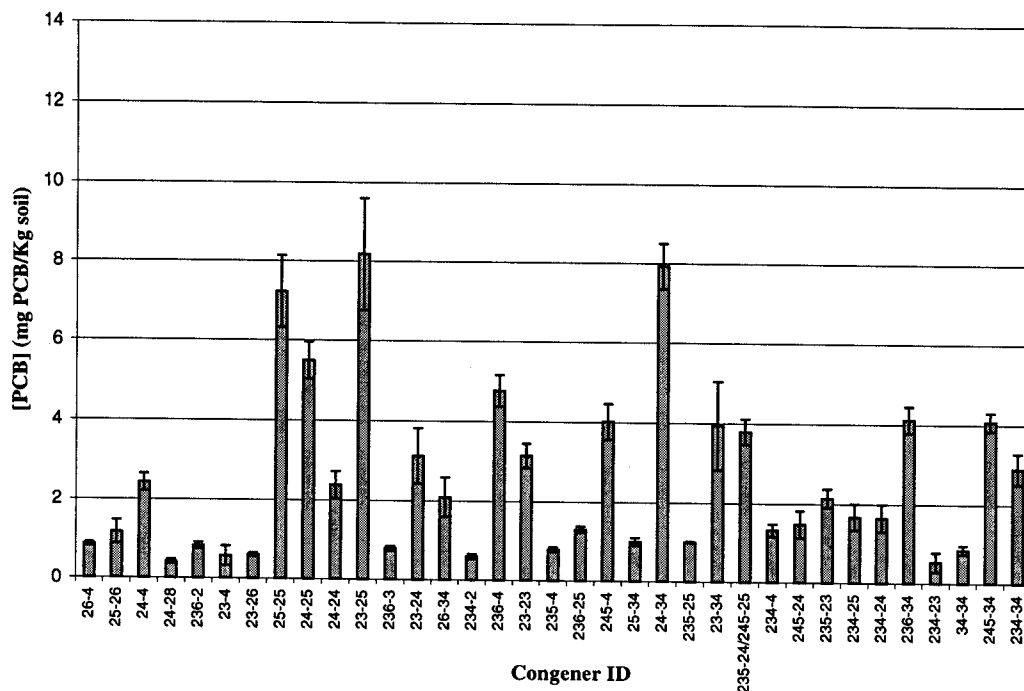
### 4.1.3 Sequential Treatment

Immediately following setup, the total average PCB concentration in Cell A1 was approximately half of the concentration observed in Cell A2. Despite this initial difference the cells demonstrated remarkably similar results throughout the experiment. Both cells exhibited stable total average PCB concentrations throughout the first 251 days of the testing period followed by a drastic PCB concentration drop (~90%) on the sixth sampling event (see Figure 11). Although this reduction coincided with the addition of biphenyl to the A1 and A2 test cells, the fact that similar reductions occurred in the other treatment conditions simultaneously indicates that the biphenyl addition was not a contributing factor to the PCB concentration reduction. The overall results from the duplicate cells A1 and A2 were very similar.

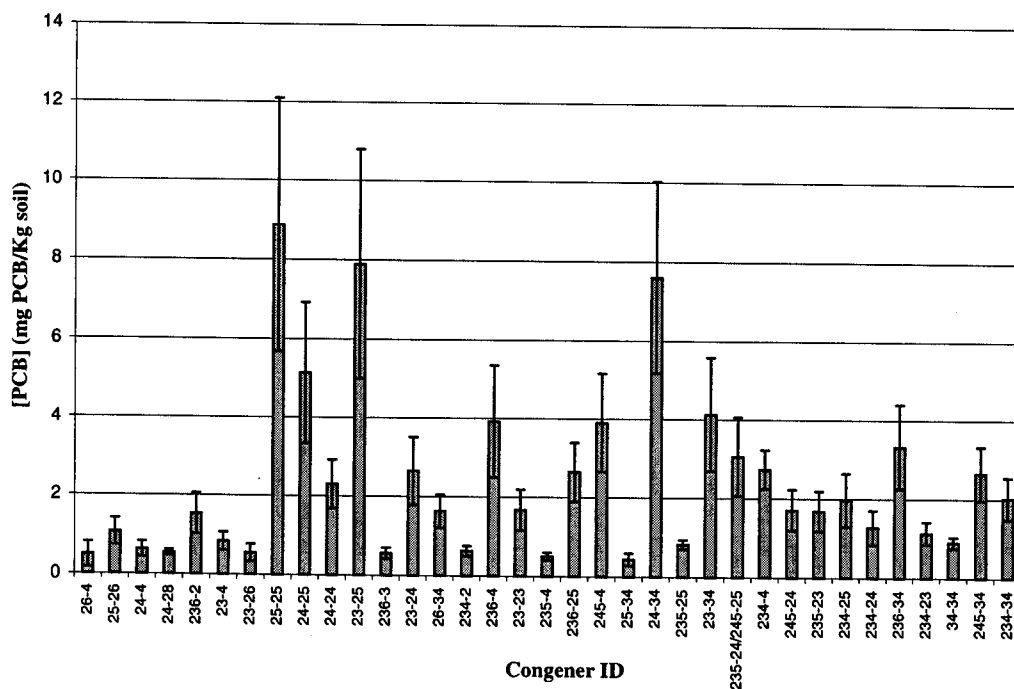


**Figure 11. Total Average PCB Concentration in Sequential Test Cells**  
(Error bars represent 95% confidence intervals [n = 5-7]. Data from the Seventh Sampling Event [Day 516] was unavailable at the time this report was written.)

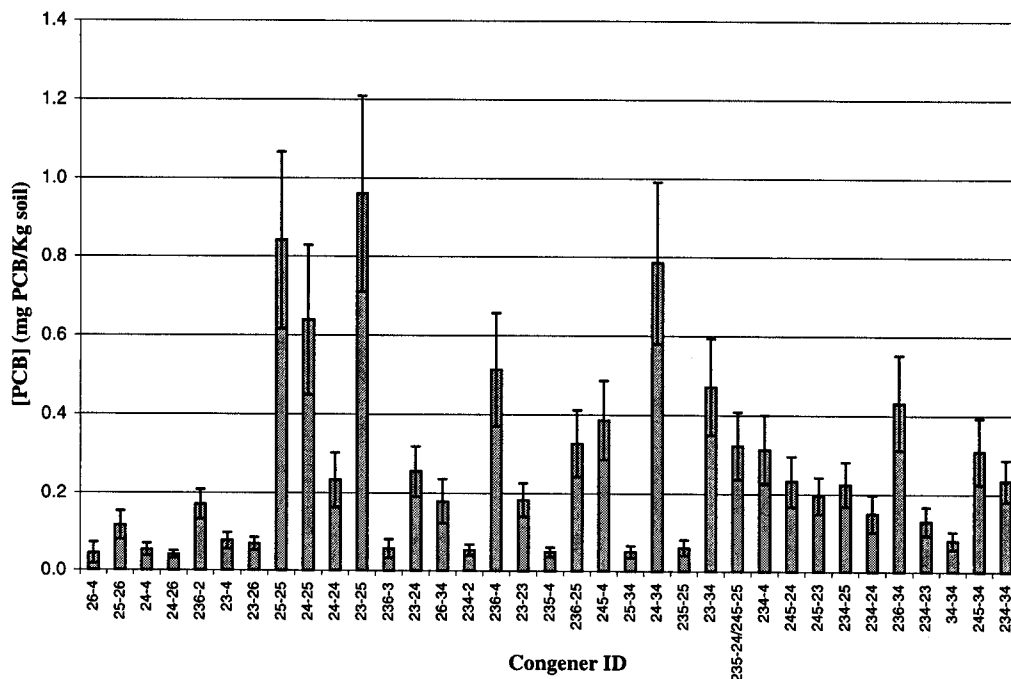
The congener fingerprint of Cells A1 and A2 were closely examined for evidence of shifting during the anaerobic phase of the treatment process, but no shifts were observed. The distribution of individual congeners remained stable throughout the experiment, indicating that no significant transformation of PCBs occurred under either anaerobic or aerobic conditions. To illustrate this finding the congener fingerprints taken at time zero and at the end of each phase (e.g., anaerobic incubation) are shown in Figures 12 through 15 for Cell A1.



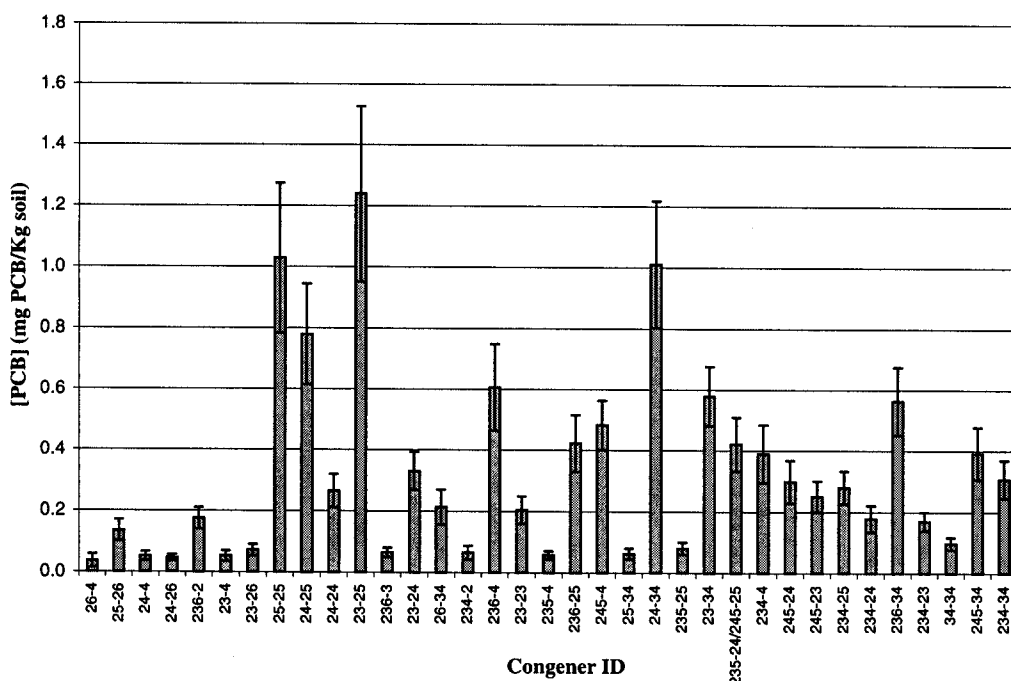
**Figure 12. Time Zero ( $t_0$ ) Congener Fingerprint for Cell A1**  
(Error bars represent 95% confidence intervals [n=6])



**Figure 13. Congener Fingerprint in Cell A1 after 202 Days of Electron-Donor Amended Anaerobic Incubation ( $t_3$ )**  
(Error bars represent 95% confidence intervals [n=5])



**Figure 14. Congener Fingerprint in Cell A1 after 476 Days (t<sub>5</sub>)**  
(Cell had undergone 202 days of electron-donor amended anaerobic incubation followed by 275 days of aerobic incubation. Error bars represent 95% confidence intervals [n=5].)



**Figure 15. Congener Fingerprint in Cell A1 at Completion of Study (t<sub>7</sub>)**  
(The total treatment time was 552 days, 202 days of electron-donor amended anaerobic incubation followed by 273 days of aerobic incubation followed by 75 days of biphenyl- and nutrient-amended incubation. Error bars represent 95% confidence intervals [n=5]).

## 4.2 Oxygen

Three oxygen probes were installed in each treatment cell at depths of 3, 6 and 9 inches to monitor the soil-gas oxygen concentration. Measurements from each probe within one cell were generally consistent indicating that soil depth did not greatly influence the availability of oxygen to microorganisms within the soil column. An additional probe was installed to measure the ambient atmospheric oxygen concentrations. This probe consistently reported oxygen concentrations ranging between 21 and 22%.

### 4.2.1 Control/Natural Attenuation Cells

The soil-gas oxygen concentrations in the control cells slowly declined from an initial level of 21% until they stabilized at about 16% (see Figure 16). This slow but steady depletion of oxygen suggests the presence of aerobic microbial activity.

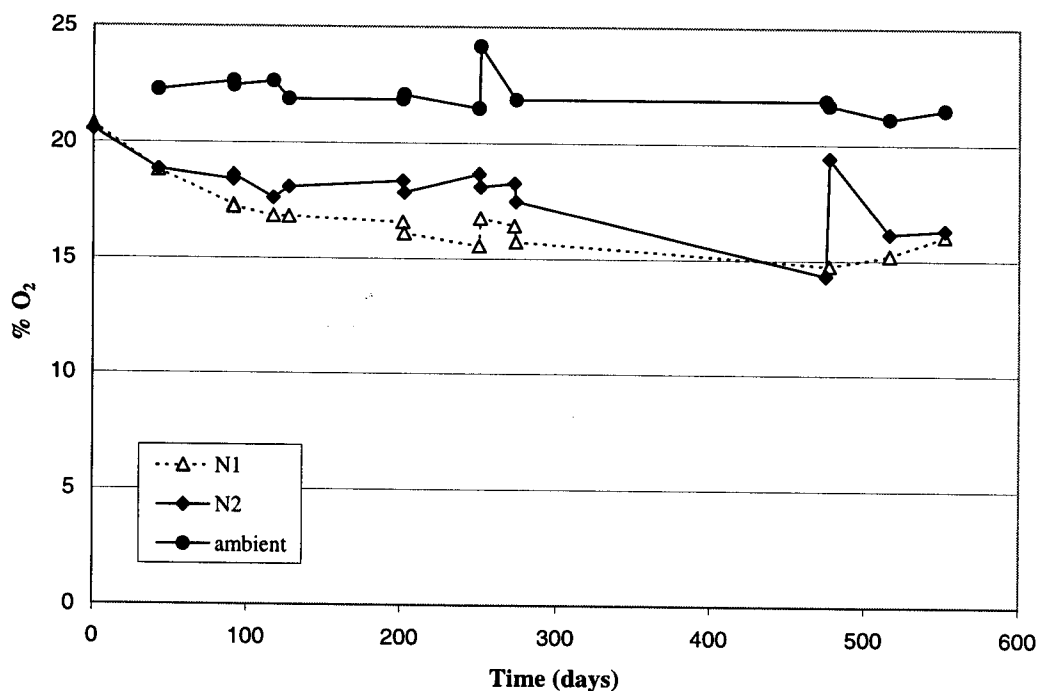
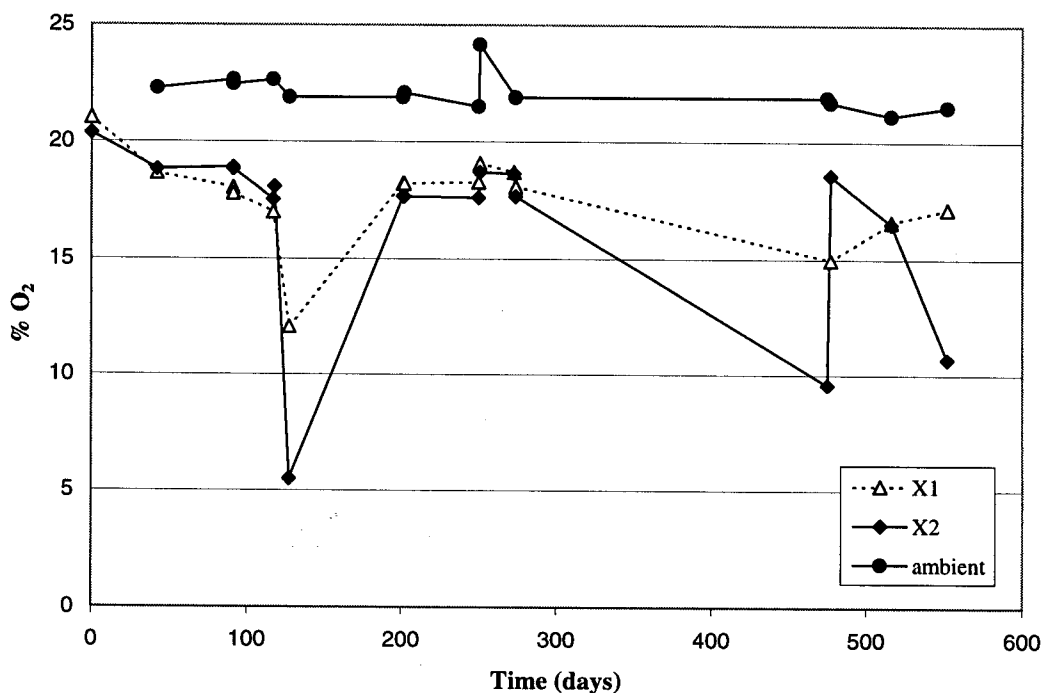


Figure 16. Average Soil-Gas Oxygen Concentrations in Control/Natural Attenuation Cells N1 and N2

### 4.2.2 X-19 Cells

The soil-gas oxygen concentration in the X-19 cells steadily decreased over the first 117 days of the experiment from 21% down to about 17% (see Figure 17). Approximately four months into the testing, heavy rains inundated Cells X1 and X2 causing a substantial decrease in oxygen concentrations. After the cells had been drained, the oxygen levels returned to previous levels (17-18% O<sub>2</sub>).



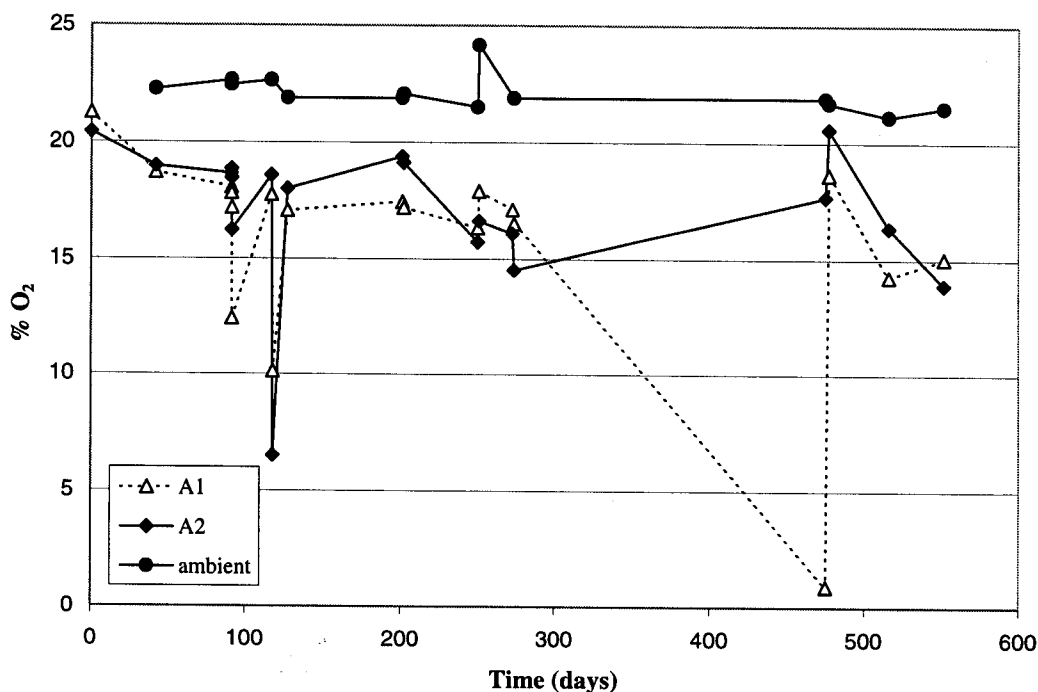
**Figure 17. Average Soil-Gas Oxygen Concentrations in X-19 Treated Cells X1 and X2**  
(Both cells were found flooded with water on Day 117. Cell X2 was found flooded again on Day 475.)

### 4.2.3 Sequential Cells

Efforts to control the soil-gas oxygen levels in sequential cells met with very limited success. The first 202 days of treatment in the sequential cells was intended to be anaerobic, but as shown in Figure 18 soil-gas oxygen levels remained between 17 and 18% for the majority of this period. Soil-gas oxygen levels were only depressed for brief periods (<10 days), which occurred immediately following feeding events. Cells were fed the acetate/butyrate mixture upon setup (Day 0), after 3 months (Day 91) and again after 4 months (Day 117). Collected oxygen data cannot verify that the cells were ever completely anoxic, but measurements taken only two hours after a feeding event show a 30% drop in oxygen concentration. This suggests that the electron donors, acetate and butyrate, were consumed very quickly causing a significant but relatively short depletion of oxygen.

The aeration of sequential test cells during the aerobic phase of the treatment did not appear to effectively raise soil-gas oxygen levels above those observed in the control/natural attenuation cells. Oxygen levels typically ranged from 13.9% to 20.6%. At one point late in the experiment the oxygen concentration in

Cell A1 dropped to less than 1%. This resulted from the cell becoming flooded with rainwater. Once the cell was drained, oxygen levels rebounded quickly.



**Figure 18. Average Soil-Gas Oxygen Concentrations in Sequentially Incubated Cells A1 and A2**  
(The anaerobic phase ran from Day 0 until Day 202. The cells were fed an acetate and butyrate solution on Days 0, 91, and 117. Cell A1 was discovered flooded on Day 475. Biphenyl and nutrients were added to both cells on Day 476.)

### 4.3 Acetate and Butyrate

An aqueous solution containing 100g of potassium acetate and 100g of sodium butyrate was added to the sequential testing cells during the anaerobic phase of the treatment process. Levels of acetate and butyrate were tracked by analyzing collected soil cores in the laboratory.

The resulting concentration increase of acetate and butyrate was estimated to be approximately 260 mg/Kg based on the mass of soil in the cells. (The mass of soil was calculated from an assumed bulk density of 1.5 g/cm<sup>3</sup> and an approximate soil volume of 9 ft<sup>3</sup>.)

Cells were fed the acetate/butyrate solution on Days 0, 91 and 117.

Although the concentration of both acetate and butyrate at time zero should have been about 260 mg/Kg, the concentration in time zero core samples, which were collected five hours after feeding, were considerably lower. Average acetate concentrations of 44.6 mg/Kg and 63.5 mg/Kg were found in Cell A1 and A2, respectively. A similar but less dramatic decrease was observed in the butyrate concentrations. Butyrate concentrations had dropped from an estimated 260 mg/Kg to 154 mg/Kg in Cell A1 and 152 mg/Kg in Cell A2.

The second round of soil samples was collected 42 days later. Despite the apparent rapid utilization of acetate inferred from the first round of samples, acetate was still present in the second round. The level had dropped to 8.2 mg/Kg in Cell A2, but surprisingly the level in Cell A1 had not changed significantly. Butyrate was not detected in these or any subsequent samples.

**Table 3. Average Acetic Acid and Butyric Acid Concentrations in Sequential Cells**

Date	Time (Days)	Average Acetic Acid Concentration (mg/Kg of soil)		Average Butyric Acid Concentration (mg/Kg of soil)	
		Cell A1	Cell A2	Cell A1	Cell A2
Jul-15-98	0	260 <sup>a</sup>	260 <sup>a</sup>	260 <sup>a</sup>	260 <sup>a</sup>
Jul-15-98	0.5	44.6	63.5	154.0	152.4
Aug-26-98	42	49.8	8.2	ND	ND
Oct-14-98	91	260 <sup>a</sup>	260 <sup>a</sup>	260 <sup>a</sup>	260 <sup>a</sup>
Nov-9-98	117	ND	ND	ND	ND
Nov-9-98	117.5	260 <sup>a</sup>	260 <sup>a</sup>	260 <sup>a</sup>	260 <sup>a</sup>
Feb-2-99	202	ND	ND	ND	ND
Mar-23-99	251	ND	ND	ND	ND

(a) Estimated concentration.

ND - not detected.

#### 4.4 Nitrate and Sulfate

The electron acceptors nitrate and sulfate were monitored during the anaerobic phase of the sequential treatment process.

Nitrate levels ranged from below detection (<1 mg/Kg) to a high around 6 mg/Kg in both cells. No trend is evident in the data. The addition of electron-donating substrates did not appear to affect nitrate levels.

Sulfate measurements in Cell A1 bounced around the average concentration of 60.1 mg/Kg. Values ranged from 46.5 mg/Kg to 75.9 mg/Kg, but no trend was evident. Sulfate measurements in Cell A2 had an overall average of 49.0 mg/Kg, and ranged between 34.5 and 64.3 mg/Kg. Unlike Cell A1, the sulfate values in Cell A2 increased steadily throughout the incubation.

**Table 4. Average Nitrate and Sulfate Concentrations in Sequential Cells**

Date	Time (Days)	Average NO <sub>3</sub> Concentration (mg/Kg of soil)		Average SO <sub>4</sub> Concentration (mg/Kg of soil)	
		Cell A1	Cell A2	Cell A1	Cell A2
Jul-15-98	0	ND	ND	59.0	34.5
Aug-26-98	42	4.2	2.2	75.9	39.8
Nov-9-98	117	ND	ND	58.1	46.7
Feb-2-99	202	2.2	3.3	46.5	59.9
Mar-23-99	251	5.9	6.0	60.8	64.3

ND - Not detected. (Detection limit = 1.0 mg/Kg)

#### 4.5 pH

The sandy soil used in this experiment was slightly alkaline with natural pH values around 8.3. Addition of the X-19 amendment caused a small decrease in the pH; conversely, adding the weak bases potassium acetate and sodium butyrate to the sequential cells caused the pH to increase. While the pH in the control/natural attenuation and X-19 treated cells remained fairly constant after setup, the pH in the sequential cells increased after every addition of acetate and butyrate (see Table 5). Acetate and butyrate were added on 15 July 1998, 14 October 1998, and 9 November 1998. The pH in sequential cells continued to rise until a pH adjustment was made on 3 November 1999.

**Table 5. Average pH in Individual Treatment Cells**

Date	Time (Days)	Average pH					
		Cell N1	Cell N2	Cell X1	Cell X2	Cell A1	Cell A2
Jul-15-98	0	8.34	8.30	8.08	8.02	8.85	9.00
Aug-26-98	42	8.22	8.14	8.05	8.03	9.14	8.99
Nov-9-98	117	8.08	8.13	8.15	8.25	9.92	9.72
Feb-2-99	202	8.13	8.09	8.13	8.00	9.79	9.68
Mar-23-99	251	8.09	8.07	8.10	8.12	10.25	10.15
Nov-3-99	476	6.94	6.88	6.89	6.63	7.49	7.45
Dec-13-99	516	NM	NM	NM	NM	NM	NM
Jan-18-00	552	NM	NM	NM	NM	NM	NM

NM – not measured.

#### 4.6 Soil Moisture

Soil moisture measurements were taken to ensure that sufficient moisture existed within the test cells to support microbial activity. Table 6 outlines the average soil moisture observed in each test cell during soil sampling events, but the values shown can be difficult to interpret without a knowledge of the soil type. The soils in cells N1, N2, A1, and A2 were sandy soils, which may have had a soil moisture content of about 17% at saturation, assuming a bulk density of 1.5g/cm<sup>3</sup> (dry weight) and a porosity of 30%. The X-19 treated cells contained a mixture of 67% sandy soils and 33% peat (bulk density 0.8 g/cm<sup>3</sup>). Peat is the primary constituent in X-19. The percent soil moisture at saturation in these cells would have been considerably higher at around 21%, assuming a bulk density of 1.3 g/cm<sup>3</sup> (dry weight) and a porosity of 35%.

The soil moisture percentages for each cell ranged considerably from almost saturated to relatively dry. Not evident in the data is the differential moisture profile observed at varying depths. Not surprisingly, the surface and shallow layers of soil dried more quickly than the soils at the bottom of each cell. It was not unusual for the top soil layer to be dry while the bottom layer was essentially saturated.

#### 4.7 Temperature

Table 7 shows the temperature measurements taken in all six cells over the first eight months of the experiment. Measurements were taken during the day with a temperature probe inserted about 4 inches into the soil. The seasonal variation of temperature is evident in the data, but the range of temperatures is relatively narrow as would be expected at a location so near the ocean. Temperatures ranged from 87.3 °F in July to 61.8 °F in March.

**Table 6. Average Soil Moisture in Individual Treatment Cells. R.S. Kerr Laboratory Data**

Date	Time (Days)	Average Soil Moisture (%)					
		Cell N1	Cell N2	Cell X1	Cell X2	Cell A1	Cell A2
15-Jul-98	0	17.4	12.4	19.0	20.5	14.8	12.6
26-Aug-98	42	6.3	4.1	14.7	17.0	10.0	10.9
9-Nov-98	117	1.9	0.6	7.2	7.3	3.1	2.9
2-Feb-99	202	8.2	3.2	17.9	23.4	18.9	15.5
23-Mar-99	251	5.1	3.0	12.5	13.4	11.3	9.3
3-Nov-99	476	16.3	14.1	21.8	21.7	15.0	12.8
13-Dec-99	516	7.1	9.4	13.5	14.9	10.2	14.5
18-Jan-00	552	5.5	7.1	11.7	12.8	10.2	12.3

**Table 7. Field Measurements of Temperature in Individual Treatment Cells**

Date	Time (Days)	Temperature (°F)					
		Cell N1	Cell N2	Cell X1	Cell X2	Cell A1	Cell A2
15-Jul-98	0	80.4	81.3	81.3	81.5	83.5	82.0
26-Aug-98	42	86.9	86.7	86.2	85.6	86.9	87.3
14-Oct-98	91	77.9	79.3	78.6	79.7	79.9	81.7
9-Nov-98	117	71.8	73.9	70.2	73.0	73.9	76.8
1-Feb-99	201	67.6	67.6	68.0	67.6	68.5	68.9
23-Mar-99	251	61.8	63.2	62.7	62.9	62.6	65.6

#### 4.8 Chloride

Soil samples were initially analyzed for chloride content in an effort to observe increases resulting from dechlorination reactions. It was soon discovered that sample variability would mask the subtle changes resulting from PCB dechlorination, so the analysis was discontinued.

Chloride levels in the natural attenuation and sequential cells were similar, ranging between 9.8 and 70.8 mg/Kg. Conversely, the X-19 cells had a considerably higher concentration with chloride ranging from 452 to 2,305 mg/Kg. The higher concentrations observed in the X-19 cells result from the concentration of chloride initially present in the X-19 product.

No trends in the data were observed.

**Table 8. Average Chloride Concentration in Individual Treatment Cells**

Date	Time (Days)	Average Chloride Concentration (mg/Kg soil)					
		Cell N1	Cell N2	Cell X1	Cell X2	Cell A1	Cell A2
15-Jul-98	0	10.4	14.7	1555	1970	15.0	10.2
26-Aug-98	42	9.8	10.9	1710	2305	20.5	14.0
9-Nov-98	117	33.3	70.8	978	452	55.1	55.3
2-Feb-99	202	11.7	15.9	1225	902	15.9	22.2
23-Mar-99	251	22.0	18.0	1495	1510	16.1	20.1

## 5.0 CONCLUSIONS

The objective of this study was to assess the potential effectiveness of three bioremediation techniques for treating PCB-contaminated soils at Air Force sites. The three techniques, natural attenuation, sequenced anaerobic/aerobic incubation and addition of the commercially available microbial amendment X-19, were tested on PCB-contaminated soils from Cape Canaveral Air Station. The PCB-contaminant profile was a combination of tri, tetra, and pentachlorobiphenyls, which probably resulted from the weathering of Aroclor 1248.

Results from the 18-month field study indicate that the three bioremediation techniques tested were ineffective at treating the PCB contamination in Cape Canaveral soils. Although data showed a significant decrease in the total average PCB concentration in each treatment condition, other evidence suggests that the treatments were not responsible for this reduction. Consider that each treatment exhibited a similar level of mass reduction simultaneously and no shifts in the congener fingerprints were observed. The most probable cause for such an observation is an analytical inconsistency. Although not confirmed by the laboratory, it is possible that data from the first five sampling events was corrected based on the recovery of an internal standard and that data from the last three sampling events was not. This would explain the behavior of the data and suggest that PCB levels were stable throughout the study. More detailed discussions of results from each treatment condition are provided below.

### 5.1 Natural Attenuation

The natural attenuation/control cells were set up to assess the background rate of PCB biodegradation in native soils. These cells were not expected to demonstrate significant changes in the PCB profile, but a significant reduction in the total average PCB mass was observed on the sixth sampling event. The observed mass reduction was later corroborated by the eighth sampling event. This surprising result prompted a thorough analysis of the congener fingerprint data to determine which congeners were being degraded. Surprisingly, a similar level of mass reduction was observed in each of the congeners, indicating that the preferential degradation of less-chlorinated congeners, which would be expected under aerobic conditions, was not occurring. In addition, similar mass reductions were observed simultaneously in the X-19 and sequentially treated test cells. The combination of these two observations suggests that the observed mass reductions were probably the result of an analytical inconsistency rather than biological activity. Based on these findings it was concluded that the PCB profile within the soils was stable and no significant changes in PCB mass content or congener composition were actually occurring.

The lack of PCB biodegradation was not due to a lack of microbial activity. Oxygen levels in the natural attenuation/control cells were about 5% lower than ambient levels throughout most of the demonstration, suggesting that aerobic microbial activity was occurring. In addition, the pH, moisture content and temperature remained within a range capable of supporting biological activity.

### 5.2 Treatment with X-19

The commercially available microbial amendment X-19 was tested in PCB-contaminated soils from Cape Canaveral to assess claims that the product could effectively degrade PCBs when used according to manufacturer's specifications. The results of this study do not support these claims. The only apparent decrease in PCB concentrations attributable to the addition of X-19 was due to the dilution of the soil with the X-19. This occurred during setup when the product was mixed with the soil at a 2:1 ratio and resulted in a lower initial PCB concentration.

Depressed soil-gas oxygen concentrations in the X-19 amended test cells suggest that aerobic microbial activity was present. On two occasions heavy rainfall caused flooding in the test cells and consequently, oxygen levels decreased significantly for a relatively brief period. Once the cells were drained, oxygen levels quickly returned to pre-flooding levels; no adverse effects were observed as a result of the flooding.

The moisture content in the X-19 treated test cells ranged from completely saturated (22% moisture by weight) to about 33% of saturation (7% moisture by weight). Based on the data it seems unlikely that microbial activity was ever inhibited by low moisture content.

The pH and temperature of the soils in the X-19 treated test cells remained within ranges suitable for microbial activity.

### **5.3 Sequential Treatment**

A series of biological incubations were used in sequence in an effort to stimulate biological destruction of PCBs in Cape Canaveral soils. The first of three phases was an anaerobic incubation amended with electron-donating substrates. Phase two was simply an aerobic incubation. The third and final phase was an aerobic incubation amended with biphenyl, designed to promote the production of 1,2 dioxxygenase and accelerate the aerobic oxidation of PCBs.

#### **5.3.1 Anaerobic Incubation**

The first phase attempted to promote reductive dechlorination of PCBs by amending the test cells with the electron-donating substrates acetate and butyrate and incubating anaerobically. After three electron-donor-feeding events and 202 days of anaerobic incubation, no significant reduction in the total average PCB concentration or shifts in the congener fingerprint were observed.

Soil-gas oxygen concentration measurements taken during the anaerobic phase showed that attempts to maintain anoxic conditions within the test cells were unsuccessful. Although oxygen levels were rapidly depleted after the addition of acetate and butyrate, they rebounded within 10 days. The mass of electron donor added was approximately 10 times that required to completely deplete each cell of oxygen assuming the test cells initially contained 2.7 ft.<sup>3</sup> of air (30% porosity). This indicates that the test cell seals were not airtight so acetate and butyrate were consumed very quickly as air diffused back into the cells. Consequently, the highly reducing environment required to reductively dechlorinate PCBs was not maintained for long periods of time. The difficulties experienced excluding oxygen at this relatively small scale would be significantly more challenging for a full-scale operation. One alternative would be to submerge the contaminated soils in water to slow the diffusion of oxygen, but this would require the disposal of potentially large quantities of contaminated water.

The alternative electron acceptors nitrate and sulfate were analyzed to track changes in the terminal electron-accepting process within each cell. Nitrate concentrations remained at or near the detection limit during the anaerobic incubation. Sulfate levels remained fairly stable at around 50 ppm. This provides a further indication that the low redox potential environment required to reductively dechlorinate PCBs was not achieved.

Collected soil cores were analyzed for acetate and butyrate to track electron-donor utilization. Only the first set of soil cores, which were collected approximately 5 hours after the first feeding, contained detectable amounts of acetate and butyrate. This illustrates the rate of electron donor consumption and corroborates the supposition that oxygen depletion resulted from electron donor oxidation.

Each addition of acetate and butyrate caused an increase in the pH in the sequential test cells. The pH rose to a high of 10.25 before the pH was adjusted with hydrochloric acid. The effect this relatively high pH had on microbial activity is not known, but soil-gas oxygen concentrations were consistently below ambient levels, suggesting that aerobic microbial activity continued to occur.

The moisture content and temperature of the soils in the sequentially treated test cells remained within ranges suitable for microbial activity during the anaerobic incubation.

Though discouraging, these results are inconclusive due to difficulties experienced in achieving and sustaining the low redox potential environment required to initiate reductive dechlorination reactions. Additional engineering controls may have been successful at sustaining lower redox potentials and therefore promoting dechlorination of the PCB profile found in the Cape Canaveral soils, but the cost associated with such a system would probably not justify the effort.

### **5.3.2 Aerobic Incubation**

The second phase of the sequential treatment process consisted of ventilating the test cells with ambient air to stimulate the aerobic oxidation of PCBs in the soil. Although mechanical difficulties prevented continuous ventilation of the cells, soil-gas oxygen levels remained relatively high at approximately 17% O<sub>2</sub>. After 273 days of aerobic incubation, no significant reduction in the total average PCB concentration or shifts in the congener fingerprint were observed. These findings demonstrate that the PCB profile encountered in these soils is resistant to aerobic biodegradation.

The pH, soil moisture and temperature of the test cells remained within ranges suitable for microbial activity throughout the aerobic incubation.

### **5.3.3 Biphenyl-Amended Aerobic Incubation**

The third phase in the sequential treatment process included amending the sequential test cells with biphenyl and nutrients and ventilating with tanks of compressed air. After 76 days of biphenyl-amended aerobic incubation, no significant reduction in the total average PCB concentration or shifts in the congener fingerprint were observed despite the fact that the biphenyl amendment was effectively degraded. The degradation of biphenyl indicates that aerobic microbial activity was present in the test cell but failed to act on the PCB contamination, further suggesting that this particular PCB profile is highly resistant to aerobic biodegradation in these soils.

The pH, soil moisture and temperature of the test cells remained within ranges suitable for microbial activity throughout the biphenyl-amended aerobic incubation.

APPENDIX A  
Laboratory-Based Analytical Method  
Standard Operating Procedures

RSKSOP-177  
Revision No. 0  
Date: 07/17/95  
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Dennis D. Fine

## STANDARD OPERATING PROCEDURE

### QUANTITATION OF PHENOLS AND ALIPHATIC AND AROMATIC ACIDS AS PENTAFLUOROBENZYL ETHERS OR ESTERS BY NEGATIVE ION CHEMICAL IONIZATION GAS CHROMATOGRAPHY/MASS SPECTROSCOPY

#### I. Disclaimer:

This Standard Operating Procedure has been prepared for the use of the Subsurface Protection and Remediation Division of the U.S. Environmental Protection Agency and may not be specifically applicable to the activities of other organizations. THIS IS NOT AN OFFICIAL EPA APPROVED METHOD. This document has not been through the Agency's peer review process or ORD clearance process.

#### II. Purpose: (Scope and Application)

This method applies to the confirmed identification and quantitation of phenols and aliphatic and aromatic acids in soil and groundwater using negative ion chemical ionization (NICI) gas chromatography/mass spectrometry (GC/MS). Phenols and acids are extracted from soil or groundwater samples and then are derivatized with pentafluorobenzylbromide to form ethers and esters. Table I lists 59 phenols and acids determined by this method. Calibration curves established for the method range from 5 to 1000 ppb (or ng/ml) of derivative injected. Recoveries of phenols and acids spiked in water blanks at 50 ppb ranged from 88% to 131%. This does not include  $C_{24}$  aliphatic acids which have extraction recoveries below 40%. Recoveries for phenols and acids spiked in contaminated soil at 167 ng per g soil ranged from 61% to 91%.

Procedures written for the Finnigan 4615 system allow automation of sample injection and unassisted control of the GC/MS system. Sample complexity and computer memory limit the throughput of the method. With moderately complex matrices up to 36 samples can be analyzed within 24 hours. Data archival is required after the analysis of about 125 samples and standards.

Only supervised analysts or analysts experienced in the use of gas chromatography/mass spectrometry and in the interpretation of chromatograms and mass spectra should use this method.

### III. Summary of Method:

A 100 ml water sample is acidified to pH 2.0 using sulfuric acid. Twenty-five g of sodium chloride is added and the water is extracted with methylene chloride. Pentafluorobenzyl ethers and esters are formed by adding acetonitrile, pentafluorobenzyl bromide and potassium carbonate to an aliquot of the extract. The internal standard is deuterated benzoic acid. For soil samples, the phenols and acids are neutralized and extracted with 10% sodium phosphate in acid free water. The aqueous extract of the soil is extracted in the same manner as the water sample. NICI GC/MS is used to identify and quantify the derivatized acids or phenols. NICI spectra of PFB derivatives are characterized by carboxylate or phenoxylate ions which correspond to the molecular weight of the acid or phenol less the acidic proton. Low picogram sensitivity of the PFB derivatives is due to the low chemical background present in NICI spectra.

### IV. Reference:

1. Operating Manuals/Applications Software for Finnigan 4500 GC/MS, Finnigan MAT, San Jose, CA.
2. Hewlett Packard 7673 Auto Injector Reference Manual, Hewlett Packard, Avondale, PA.
3. Determination of Carboxylic Acids and Phenols in Water by Extractive Alkylation Using Pentafluorobenzylation, Glass Capillary GC and Electron Capture Detection, E. Fogelqvist, B. Josefsson, C. Roos, JHRC&CC, 570 (1980) 568-574.
4. Determination of Polar Pesticides by Phase-Transfer Catalyzed Derivatization and Negative-Ion Chemical Ionization Gas Chromatography-Mass Spectrometry, H. Meiring, G. den Engelsman and A. de Jong, J. Chromatog., 644 (1993) 357-365.

V. Procedure:

A. Liquid-Liquid Extraction of Phenols and Aliphatic/Aromatic Acids.

Deliver 100 ml of water sample to a dried, silanized 125 ml separatory funnel. If applicable add spike standards to the sample. Adjust the pH of the water to 2.0 using 1:1 concentrated sulfuric acid. For an unpreserved water blank, add ten drops of acid. For 100 ml of water sample preserved with sodium phosphate, tribasic dodecahydrate, add twenty drops of acid. Next add 25 g of sodium chloride to the separatory funnel and swirl the liquid to dissolve the salt.

Extract the water sample four times with 5 ml aliquots of acid free methylene chloride. Collect the methylene chloride extracts in silanized 40 ml VOA vials. Record the total extract volume.

To remove acids from methylene chloride and other solvents, add 10 g of Celite Micro-Cel T-49 to one liter of GC/MS grade solvent. Stir this mixture for one hour, allow the solid to settle and filter the liquid through a Millipore organic or aqueous filter pad using a Millipore vacuum apparatus.

Silanize the glassware with Sylon CT (Supelco, Inc.) for 1 hour and then rinse three times with methylene chloride before use.

B. Extraction of Phenols and Aliphatic/Aromatic Acids from Soil.

Deliver 20 ml of 10% wt. by vol. sodium phosphate, tribasic dodecahydrate in water to 30 g of soil sample contained in a 40 ml VOA vial. Shake the vial for 10 minutes and then centrifuge it at 1500 RPM. Pour the aqueous layer off the soil into a 100 ml graduated cylinder. Repeat the extraction two more times. Add the measured volume of aqueous extract to a silanized 125 ml separatory funnel. Add more 10% wt. by vol. sodium phosphate, tribasic dodecahydrate to bring the total volume of water in the separatory funnel up to 100 ml. Continue with the soil extracts in exactly the same manner as described in section A. For soil samples containing high organic material, liquid/liquid extraction may take longer if emulsions occur.

C. Phenol/Acid Derivatization to Form PFB Ethers and Esters.

Dry about 50 g of potassium carbonate overnight in a muffle furnace at 600°C. Deliver 200  $\mu$ l of the methylene chloride extract of the aqueous or soil sample to a 2 ml screw cap vial containing 2.5 mg of potassium carbonate. Use a Teflon faced septum in the screw cap. Next add 790  $\mu$ l of acid free acetonitrile, 10  $\mu$ l of 100 ppm benzoic acid-d<sub>5</sub> in acetonitrile and 10  $\mu$ l of pentafluorobenzyl bromide to the vial. Benzoic acid-d<sub>5</sub> is the internal standard for the analysis. Momentarily place the vials in a ultrasonic bath to free the potassium carbonate from the bottom of the vial. Tighten the screw caps and heat the vials in an oven at 60°C for 2 hours. Remove the vials from the oven and add 500  $\mu$ l of 0.1M hydrochloric acid. Shake the vials for 30 seconds and deliver 200  $\mu$ l of the top organic layer to the liner insert of a 2 ml crimp cap autosampler vial.

D. Autoinjector Operation.

Place the standards, blanks or samples into the Hewlett Packard 7673 autosampler tray. Although the tray will hold up to 100 vials, analysis queues usually contain about 30 standards, blanks and samples. Ensure that the syringe wash vials are filled with methylene chloride. Ensure that the controller power switch is on and that the injector has the following settings:

Sample size	0.5 $\mu$ l
Injection #/vial	1
Sample Pre-wash	2
Solvent Post-wash	6

E. GC/MS Preparation and Operation.

(1). Replace the injection port septum if ten or more injections have been made. Do this at an oven temperature of 40°C. Condition the capillary column at 300°C for 20 minutes when the gas chromatograph has been idle for over 10 hours.

(2). Check that the helium carrier gas linear velocity has not changed. Set the oven temperature to 80°C and remove the HP 7673

autoinjector from the top of the injector. Inject 1.0  $\mu$ l of air into the injection port. For a desired linear velocity of 36 cm/s through the 60 meter column with 0.25 mm i.d., adjust the column pressure until the time from injection to detection of the air spectrum is 167 seconds. Set the split and septum purge flows to 20 and 2 ml/min, respectfully.

(3). Before tuning the mass spectrometer for negative ions, the instrument is calibrated for electron impact (positive ions) using perfluorotributylamine (FC43). Turn on the calibration gas switch to allow FC43 into the ion source and adjust the quadrupole offset, offset program, extractor voltage, lens voltage and entrance voltage of ion source to obtain an electron ionization spectrum of FC43 which will satisfy the EPA tuning criteria for decafluorotriphenylphosphine (DFTPP). Use the procedure CALGAS, as listed on page 96 to automatically set up the scan profile, acquire the calibration, update the calibration spectrum file and print out the calibration spectrum. Figures 1 and 2 show the desired mass spectrum and spectral list of FC43.

(4). Check that the mass spectrometer meets the EPA tune criteria by injecting 1.0  $\mu$ l of 50 ng/ $\mu$ l decafluorotriphenylphosphine (DFTPP) in methylene chloride using the autoinjector. Place a vial containing this solution in the sample tray and enter the procedure DFTPP as provided on page 97 on the console. Use the oven temperature program as provided on page 99. Figures 3 - 6 show a typical TIC chromatogram showing the retention and peak area of DFTPP, the mass spectrum of DFTPP, a mass spectral list and a tuning criteria print out. Should the mass spectrum of DFTPP fail to meet the tuning criteria as shown in Figure 6, see possible corrective action in Section XI.

(5). For NICI GC/MS, place a chemical ionization ion volume in the ion source block of the Finnigan 4615 GC/MS. Turn on the valve to allow ultra high purity methane to enter the source. Adjust the methane needle valve until the ionizer pressure reaches 0.40 torr. With the ionizer at this pressure, the high vacuum pressure indicates  $1.0 \times 10^{-5}$  torr. Change the ionization control from QEM to Local. Set the scan mode to negative and the ionizer mode to CI. Tune the mass spectrometer for negative ions by using the calibration gas, FC-43. Adjust the negative ion controls for the ion offset, extractor, lens, and quadrupole entrance voltages to obtain good peak shape for ions 414 and 633 m/z and a relative intensity near 100:44:12 for ions 633, 414 and 127 m/z. Use the CALGAS procedure and the calibration table,

NIFC43.CT to obtain a calibration for negative ions. Figures 7 - 8 show a NICI mass spectrum and spectral list of the calibration gas.

(6). If the system was previously calibrated using standard mixtures, run system blanks to check for artifacts and check standards to confirm stability of the response curves. If the system was not calibrated or if response curves have failed due to system changes, prepare new calibration curves.

VI. Finnigan 4615 Gas Chromatograph/Mass Spectrometer  
NICI Operating Conditions:

(1) Gas Chromatograph: Finnigan 9611

Column: J&W DB5-MS 60 m x 0.25 mm x 0.25  $\mu$ m

Air Dead Time: 167 sec (80 °C)  
Carrier Linear Velocity: 36 cm/sec  
Helium Pressure: 29 psi  
Split Flow: 20 ml/min  
Septum Sweep: 2 ml/min

Injector Temperature: 275 °C

GC Temperature Program Conditions:

Splitless Injection Time: 1.0 min  
Initial Temperature: 50 °C  
Initial Time: 1.0 min  
Program Rate: 30 °C/min  
Temperature 2: 100 °C  
Program Rate 2: 6 °C/min  
Final Temperature: 300 °C  
Final Time: 16.5 min

(2) Mass Spectrometer: Finnigan 4615

Scan Range: 42 to 550 m/z in 0.5 sec  
Interface Oven Temperature: 275 °C  
Manifold Temperature: 90 °C  
Ionizer Temperature: 150 °C

Transfer Line Temperature:	225 °C	
High Vacuum:	1.0 x 10 <sup>-5</sup> Torr	
Ionizer Forepressure (CH <sub>4</sub> CI):	0.40 Torr	
Methane Tank 2nd Stage Pressure:	40 psi	
Inlet Forepressure:	0.01 Torr	
Manifold Forepressure:	0.04 Torr	
Ionizer Control:	Local	
Scan Mode:	Neg	
Preamp Sensitivity:	10 <sup>-7</sup> amps/volt	
Preamp Filter:	10 x100	
Emission Current:	70.0 eV	
Electron Multiplier Voltage:	-1250 volts	
Quadrupole Offset:	+4.0 volts	***
Offset Program:	Dial Set to 9:30	***
Extractor:	-1.9 volts	***
Lens Voltage:	+1.0 volts	***
Quadrupole Entrance Voltage:	-33 volts	***
Ion Conversion Dynode:	+4.9 kvolts	

\*\*\* These parameters are adjusted to achieve the proper tune criteria and actual values may not correspond to the values listed here.

(3) For more Finnigan 4615 parameters, see Figures 9 - 10.

#### VII. Analysis Profile - Calibration Standards, Quality Assurance Samples and Analysis Queues:

Prepare standard curves using a mixture containing 13 phenols, 27 aliphatic acids and 19 aromatic acids. Four sets of these standards: METI 148-237A, METI 148-233, METI 148-235 and METI 148-260B were previously prepared in high purity HPLC acetonitrile. Information concerning the preparation of these standards is contained in Mantech Environmental Technology, Inc., Laboratory Notebook 148. Each 1000 ppm stock solution was prepared by adding 100 mg of each phenol or acid to acetonitrile in a 100 ml volumetric flask. Each neat phenol or acid has a purity greater than 95%. Acetic acid was not included in the standard curves due to acetic acid artifacts in the acetonitrile. Working stock solutions were prepared by combining and diluting the four stock solutions to obtain 1, 10 and 100 ppm. These solutions are labelled: METI 148-264A, METI 148-264B and METI 148-264C. All stock and working solutions were ampuled and archived.

Prepare standards at 5, 10, 25, 50, 100, 500 and 1000 ppb using the concentrations and volumes of solvents and reagents shown in the Figure 11. Use the procedure AUTONI in Appendix II for entry of the analysis sequence into the GC/MS data system and for control of the GC/MS during the analyses. Enter sample names into a name list and accounting information for each sample name. Figure 11, 12 and 13 give examples of a sample queue, name list and accounting information. The procedure AUTONI initializes the scan profile and GC temperature program, controls the splitless injection, starts the GC program, controls the injection of the sample, starts the acquisition, turns on the filament and electron multiplier, and initializes the GC/MS before the next injection. The GC/MS system automatically stores spectral files for each of the samples and prints out chromatograms after each run. Quantitation procedures provided by Finnigan are run to identify and quantitate the compounds listed in the target compound list.

Figures 14a-zz show the chromatogram of a 100 ppb calibration standard. Peaks in these chromatograms are labelled with the corresponding phenol or acid. Confirmation of each peak was done by injecting the individual compound.

#### VIII. Quality Assurance:

Ensure quality assurance of the GC/MS analysis by running check standards, method blanks, derivative blanks, fortified method blanks and sample spikes throughout the analysis sequence. EPA certified standards containing the acidic compounds are not available as quality control (QC) standards. Determine the extraction efficiencies for each target compound by running sample spikes and fortified water blanks. Also plot a control chart for the peak area for the internal standard, benzoic acid- $d_5$ .

Examples of check standards values, extraction efficiencies and control charts are provided in Figures 15 - 18. Figures 15 and 16 show plots of concentrations determined for check standards at 10 and 100 ppb. Figure 17 shows a plot of the extraction recoveries for five 100 ml water blanks spiked at 50 ppb with the phenols and aliphatic and aromatic acids. Table II shows the data and statistics for these extractions. Figure 18 provides recoveries of four field samples which were spiked with 50 ppb of phenols and aliphatic and aromatic acids. Table III contains the data and statistics for these extractions. A

control chart of the peak areas of benzoic acid-d<sub>5</sub>-PFB is shown in Figure 19.

The criteria for confirmed identification are a library fit greater than 800 where a fit of 1000 implies a perfect fit and a change in the expected compound retention time of +/- 5 seconds. The Finnigan quantitation procedure, Target Compound Analysis (TCA), allows setting of these parameters. Should the quantitation procedure fail either of these criteria, the procedure reports "Not Found" for the target compound.

Should any of the quality control parameters fail to meet the criteria set in Table IV, check the possible corrective actions listed Section X.

#### IX. Calculations and Data Archival:

For quantitative analysis, calculate the relative peak area for the peak area of the characteristic ion of the phenol/acid PFB compound and the peak area of characteristic ion of benzoic acid-d<sub>5</sub>-PFB. Table I provides the characteristic ions which are used for quantitation. Interpolate the concentration of analytes in the injected solution from a linear regression fit of the calibration peak area ratios and the standard concentrations. Figure 20a-zz provides examples of calibration curves and response data.

When the volumes of water sample, extracts and reagents are used as described in this procedure, the concentration of analyte determined in the injected extract equals the concentration in the water sample.

Calibration, quality control and sample acquisition data are stored on magnetic tape and are archived for five years.

#### X. Corrective Actions:

A. If the tuning criteria for DFTPP cannot be met, consider the following corrective actions:

- (1). Incorrect mass identification - Recalibrate system with calibration gas, FC-43.

(2). Intensities at low end of DFTPP spectrum too high or too low - adjust Quad Offset.

(3). Natural abundance isotope ratios do not fall within criteria range - adjust extractor, lens and quadrupole entrance voltages and resolution potentiometers.

(4). If the tuning criteria cannot be met by adjustments to extractor, lens and quadrupole entrance voltages or resolution potentiometers, clean the lenses, ion volume and quadrupole rods.

B. If nonpolar compounds show loss of peak height or if acidic or basic compounds exhibit peak tailing:

(1). Replace fused silica wool in liner and/or clean the quartz injection liner.

(2). Flush the capillary with solvent according to manufacturer's instruction.

(3). Break off a short portion (about 20 cm) of the column from the end near the injector; or replace the capillary column.

C. Failure to meet quality assurance criteria listed in Table II.

(1). If acid compounds or artifacts are present in blanks, consider preparing fresh acid free solvents; reheating potassium carbonate, purchasing new reagents or resilanizing glassware.

(2). If the internal standard peak area shifts outside the control chart, determine the peak area of the internal standard from a freshly opened ampule. If the peak area is still outside the control chart, consider evaluation of the electron multiplier, filament and ion source.

(3). If calculated values of check standards are not within 20% of expected value, run freshly opened ampules of standard mixtures.

XI. Instrument Maintenance:

- (1). Change the mechanical pump oil every three months.
- (2). Clean the ion volume, lenses and quadrupole rods as needed.
- (3). Check the water level in the refrigerated cooler monthly.
- (4). Change the injector septum daily or after every 10 injections.
- (5). Archive data, quantitation, calibration and library files on tape as the hard drive becomes full or upon completion of the service request.
- (6). Backup the system disc once a month or when new application procedures are written.

XII. Safety:

Some method analytes have been tentatively classified as known or suspected carcinogens. As such, pure standard materials and stock standard solutions of these compounds should be handled in a hood. Plastic gloves, safety glasses and laboratory jackets should be worn.

Table I. Retention Time, Scan Number and Quantitation Ion for Phenols and Aliphatic/Aromatic Acid-PFB Derivatives.

Library AC.#	Compound Name	Quantitation Ion	Scan Number	Retention Time
1	ACETIC ACID - PFB	59	1136	9.47
2	PROPANOIC ACID - PFB	73	1344	11.20
3	2-METHYLPROPANOIC ACID - PFB	87	1433	11.93
4	TRIMETHYL ACETIC ACID - PFB	101	1471	12.25
5	BUTYRIC ACID - PFB	87	1560	13.00
6	2-METHYLBUTYRIC ACID - PFB	101	1658	13.82
7	3-METHYLBUTYRIC ACID - PFB	101	1684	14.03
8	3,3-DIMETHYLBUTYRIC ACID - PFB	115	1782	14.85
9	PENTANOIC ACID - PFB	101	1807	15.05
10	2,3-DIMETHYLBUTYRIC ACID - PFB	115	1837	15.30
11	2-ETHYLBUTYRIC ACID - PFB	115	1851	15.42
12	2-METHYLPENTANOIC ACID - PFB	115	1863	15.52
13	3-METHYLPENTANOIC ACID - PFB	115	1948	16.23
14	4-METHYLPENTANOIC ACID - PFB	115	1964	16.37
15	HEXANOIC ACID - PFB	115	2057	17.13
16	2-METHYLHEXANOIC ACID - PFB	129	2096	17.47
17	PHENOL - PFB	93	2108	17.57
18	CYCLOPENTANECARBOXYLIC ACID - PFB	113	2208	18.40
19	5-METHYLHEXANOIC ACID - PFB	129	2210	18.42
20	O-CRESOL - PFB	107	2272	18.93
21	2-ETHYLHEXANOIC ACID - PFB	143	2268	18.90
22	HEPTANOIC ACID - PFB	129	2306	19.22
23	M-CRESOL - PFB	107	2334	19.45
24	P-CRESOL - PFB	107	2358	19.65
25	1-CYCLOPENTENE-1-CARBOXYLIC ACID - PFB	111	2363	19.68
26	O-ETHYLPHENOL - PFB	121	2430	20.25
27	CYCLOPENTANEACETIC ACID - PFB	127	2450	20.42
28	2,6-DIMETHYLPHENOL - PFB	121	2461	20.50
29	2,5-DIMETHYLPHENOL - PFB	121	2468	20.57
30	CYCLOHEXANECARBOXYLIC ACID - PFB	127	2478	20.65
31	3-CYCLOHEXENE-1-CARBOXYLIC ACID - PFB	125	2495	20.78
32	2,4-DIMETHYLPHENOL - PFB	121	2500	20.87
33	3,5-DIMETHYLPHENOL & M-ETHYLPHENOL - PFB	121	2560	21.08
34	OCTANOIC ACID - PFB	143	2548	21.23
35	2,3-DIMETHYLPHENOL - PFB	121	2576	21.47
36	P-ETHYLPHENOL - PFB	121	2583	21.52
37	BENZOIC ACID(D5) - PFB (INT. STD.)	126	2594	21.62
38	BENZOIC ACID - PFB	121	2600	21.67
39	3,4-DIMETHYLPHENOL - PFB	121	2638	21.98
40	M-METHYLBENZOIC ACID - PFB	135	2677	22.30
41	1-CYCLOHEXENE-1-CARBOXYLIC ACID - PFB	125	2685	22.37
42	CYCLOHEXANEACETIC ACID - PFB	141	2691	22.42
43	2-PHENYLPROPANOIC ACID - PFB	149	2694	22.45
44	O-METHYLBENZOIC ACID - PFB	135	2772	23.10
45	PHENYLACETIC ACID - PFB	135	2834	23.62
46	M-TOLYLACETIC ACID - PFB	149	2857	23.80
47	O-TOLYLACETIC ACID - PFB	149	2867	23.88
48	2,6-DIMETHYLBENZOIC ACID - PFB	149	2873	23.93
49	P-TOLYLACETIC ACID - PFB	149	2886	24.05
50	P-METHYLBENZOIC ACID - PFB	135	2890	24.08
51	3-PHENYLPROPANOIC ACID - PFB	149	2953	24.60
52	2,5-DIMETHYLBENZOIC ACID - PFB	149	2974	24.78
53	DECANOIC ACID - PFB	171	3013	25.10
54	2,4-DIMETHYLBENZOIC ACID - PFB	149	3038	25.32
55	3,5-DIMETHYLBENZOIC ACID - PFB	149	3044	25.37
56	2,3-DIMETHYLBENZOIC ACID - PFB	149	3079	25.65
57	4-ETHYLBENZOIC ACID - PFB	149	3115	25.95
58	2,4,6-TRIMETHYLBENZOIC ACID - PFB	163	3116	25.97
59	3,4-DIMETHYLBENZOIC ACID - PFB	149	3176	26.47
60	2,4,5-TRIMETHYLBENZOIC ACID - PFB	163	3289	27.40

Table II. Recovery of 50 ppb Phenols and Aliphatic and Aromatic Acids Spiked into  
100 ml Blank Water.

Cmpd. No.	Compound	% Recovery of 50 ppb Spiked Into 100 ml of Blank Water					Average Recovery n=5	Sample Standard Deviation	% Relative Std. Dev.
3	2-METHYLPROPANOIC ACID - PFB	41	39	40	33	32	37	3.7	10.0
4	TRIMETHYL ACETIC ACID - PFB	104	97	100	84	77	92	10.3	11.2
5	BUTYRIC ACID - PFB	42	39	40	35	34	38	2.9	7.8
6	2-METHYLBUTYRIC ACID - PFB	96	92	95	81	78	88	8.4	9.5
7	3-METHYLBUTYRIC ACID - PFB	89	83	86	75	70	81	8.8	8.5
8	3,3-DIMETHYLBUTYRIC ACID - PFB	118	110	113	100	91	108	9.7	9.1
9	PENTANOIC ACID - PFB	103	96	98	89	85	94	6.5	8.9
10	2,3-DIMETHYLBUTYRIC ACID - PFB	124	118	118	104	96	112	10.5	9.4
11	2-ETHYLBUTYRIC ACID - PFB	121	113	115	101	94	109	9.7	8.9
12	2-METHYLPENTANOIC ACID - PFB	111	108	107	96	88	102	8.4	8.3
13	3-METHYLPENTANOIC ACID - PFB	116	109	112	101	93	106	8.2	7.7
14	4-METHYLPENTANOIC ACID - PFB	117	110	114	108	97	109	7.2	8.8
15	HEXANOIC ACID - PFB	127	122	124	118	111	120	5.8	4.8
16	2-METHYLHEXANOIC ACID - PFB	118	111	112	103	97	108	7.2	8.7
17	PHENOL - PFB	107	100	101	83	78	94	11.3	12.1
18	CYCLOPENTANECARBOXYLIC ACID - PFB	101	97	100	91	83	94	6.7	7.1
19	5-METHYLHEXANOIC ACID - PFB	120	112	118	107	98	110	7.7	7.0
20	o-CRESOL - PFB	114	109	109	92	86	102	10.9	10.6
21	2-ETHYLHEXANOIC ACID - PFB	122	115	117	108	99	113	8.0	7.1
22	HEPTANOIC ACID - PFB	129	120	126	123	115	123	4.8	3.8
23	m-CRESOL - PFB	118	109	110	94	88	103	10.5	10.2
24	p-CRESOL - PFB	113	108	109	93	88	102	10.5	10.3
25	1-CYCLOPENTENE-1-CARBOXYLIC ACID - PFB	108	102	106	97	91	101	6.1	6.1
26	o-ETHYLPHENOL - PFB	115	110	109	93	88	103	10.5	10.2
27	CYCLOPENTANECETIC ACID - PFB	119	112	115	109	101	111	8.0	5.4
28	2,6-DIMETHYLPHENOL - PFB	92	88	88	73	78	83	7.4	8.9
29	2,5-DIMETHYLPHENOL - PFB	106	102	104	87	85	97	9.1	9.3
30	CYCLOHEXANECARBOXYLIC ACID - PFB	117	111	115	106	97	108	7.1	8.5
31	3-CYCLOHEXENE-1-CARBOXYLIC ACID - PFB	113	107	110	101	95	105	6.4	6.1
32	2,4-DIMETHYLPHENOL - PFB	90	84	83	69	75	80	7.3	9.0
33	3,5-DIMETHYLPHENOL & M-ETHYLPHENOL - PFB	114	108	108	94	87	103	10.1	9.9
34	OCTANOIC ACID - PFB	132	125	129	131	123	128	3.5	2.7
35	2,3-DIMETHYLPHENOL - PFB	111	107	108	93	88	101	8.9	8.8
36	p-ETHYLPHENOL - PFB	114	107	110	91	88	102	10.8	10.8
37	BENZOIC ACID - PFB	129	120	125	118	112	121	5.9	4.9
38	3,4-DIMETHYLPHENOL - PFB	111	105	108	89	85	99	10.0	10.1
39	m-METHYLBENZOIC ACID - PFB	117	107	115	118	108	112	4.7	4.2
40	1-CYCLOHEXENE-1-CARBOXYLIC ACID - PFB	121	115	119	113	104	114	6.0	5.2
41	CYCLOHEXANECETIC ACID - PFB	124	118	122	116	109	118	5.4	4.6
42	2-PHENYLPROPANOIC ACID - PFB	128	117	123	119	110	119	5.9	4.9
43	o-METHYLBENZOIC ACID - PFB	130	123	126	118	108	121	7.8	6.4
44	PHENYLACETIC ACID - PFB	134	126	132	127	121	128	4.8	3.7
45	m-TOLYLACETIC ACID - PFB	127	119	123	130	118	123	4.8	3.7
46	o-TOLYLACETIC ACID - PFB	131	126	129	134	119	128	5.1	4.0
47	2,6-DIMETHYLBENZOIC ACID - PFB	132	120	125	109	99	117	11.8	9.9
48	p-TOLYLACETIC ACID - PFB	128	124	124	136	126	127	4.6	3.8
49	p-METHYLBENZOIC ACID - PFB	133	128	131	129	119	126	4.8	3.7
50	3-PHENYLPROPANOIC ACID - PFB	122	118	123	126	119	122	2.8	2.3
51	2,5-DIMETHYLBENZOIC ACID - PFB	128	124	125	118	111	121	6.1	5.0
52	DECANOIC ACID - PFB	121	118	122	128	119	121	3.8	3.1
53	2,4-DIMETHYLBENZOIC ACID - PFB	128	122	125	119	111	121	5.8	4.6
54	3,5-DIMETHYLBENZOIC ACID - PFB	133	129	128	127	119	127	4.3	3.4
55	2,3-DIMETHYLBENZOIC ACID - PFB	132	126	128	123	112	124	6.7	5.4
56	4-ETHYLBENZOIC ACID - PFB	136	129	132	133	128	131	3.2	2.4
57	2,4,6-TRIMETHYLBENZOIC ACID - PFB	131	124	125	112	103	119	10.0	8.4
58	3,4-DIMETHYLBENZOIC ACID - PFB	128	125	131	128	119	126	4.2	3.4
59	2,4,5-TRIMETHYLBENZOIC ACID - PFB	123	120	123	117	107	118	5.9	5.0

Table III. Recovery of 50 ppb Phenols and Aliphatic and Aromatic Acids Spiked into 100 ml of Four Different Field Samples.

Compd. No.	Compound	%Recovery of 50 ppb Spiked into 100 ml of Field Samples				Average Recovery n = 4	Sample Standard Deviation	% Relative Std.Dev.
3	2-METHYLPROPANOIC ACID - PFB	43	34	38	27	36	6.8	19.1
4	TRIMETHYL ACETIC ACID - PFB	104	87	89	87	92	8.1	8.9
5	BUTYRIC ACID - PFB	39	35	34	22	33	7.5	23.1
6	2-METHYLBUTYRIC ACID - PFB	103	86	89	25	78	34.8	46.0
7	3-METHYLBUTYRIC ACID - PFB	94	79	81	26	70	30.2	43.3
8	3,3-DIMETHYLBUTYRIC ACID - PFB	122	106	110	105	111	8.0	7.2
9	PENTANOIC ACID - PFB	106	91	94	91	95	6.9	7.3
10	2,3-DIMETHYLBUTYRIC ACID - PFB	128	110	111	109	115	8.7	7.6
11	2-ETHYLBUTYRIC ACID - PFB	123	106	109	102	110	9.2	8.3
12	2-METHYLPENTANOIC ACID - PFB	116	100	102	101	105	7.6	7.3
13	3-METHYLPENTANOIC ACID - PFB	122	105	108	106	110	8.0	7.2
14	4-METHYLPENTANOIC ACID - PFB	124	111	111	108	114	7.1	6.3
15	HEXANOIC ACID - PFB	124	112	108	107	113	7.8	6.9
16	2-METHYLHEXANOIC ACID - PFB	120	111	107	105	111	6.8	6.1
17	PHENOL - PFB	107	81	88	83	90	12.1	13.5
18	CYCLOPENTANECARBOXYLIC ACID - PFB	111	96	101	97	101	7.0	6.9
19	5-METHYLHEXANOIC ACID - PFB	121	112	109	108	113	5.9	5.2
20	o-CRESOL - PFB	117	93	99	95	101	10.9	10.8
21	2-ETHYLHEXANOIC ACID - PFB	119	124	118	103	116	9.0	7.8
22	HEPTANOIC ACID - PFB	128	123	120	117	122	4.7	3.8
23	m-CRESOL - PFB	120	96	102	98	104	10.9	10.5
24	p-CRESOL - PFB	118	90	103	56	92	26.6	29.0
25	1-CYCLOPENTENE-1-CARBOXYLIC ACID	115	103	105	106	107	5.4	5.0
26	o-ETHYLPHENOL - PFB	115	96	101	97	102	8.9	8.7
27	CYCLOPENTANEACETIC ACID - PFB	128	116	119	113	119	6.2	5.2
28	2,6-DIMETHYLPHENOL - PFB	110	85	95	92	95	10.5	11.0
29	2,5-DIMETHYLPHENOL - PFB	114	93	101	96	101	9.5	9.4
30	CYCLOHEXANECARBOXYLIC ACID - PFB	125	112	114	110	115	6.8	5.9
31	3-CYCLOHEXENE-1-CARBOXYLIC ACID -	124	106	111	108	112	8.1	7.2
32	2,4-DIMETHYLPHENOL - PFB	113	73	100	95	95	16.7	17.6
33	3,5-DIMETHYLPHENOL & M-ETHYLPHENO	115	98	100	98	103	8.5	8.2
34	OCTANOIC ACID - PFB	122	126	123	120	123	2.2	1.8
35	2,3-DIMETHYLPHENOL - PFB	118	94	101	97	103	10.8	10.5
36	p-ETHYLPHENOL - PFB	114	94	101	93	100	9.7	9.6
37	BENZOIC ACID - PFB	115	109	113	94	108	9.5	8.8
38	3,4-DIMETHYLPHENOL - PFB	116	89	97	95	100	11.6	11.7
39	m-METHYLBENZOIC ACID - PFB	128	122	135	89	118	20.4	17.3
40	1-CYCLOHEXENE-1-CARBOXYLIC ACID -	129	120	118	114	120	6.3	5.3
41	CYCLOHEXANEACETIC ACID - PFB	129	125	126	122	126	2.9	2.3
42	2-PHENYLPROPANOIC ACID - PFB	131	128	131	124	129	3.2	2.5
43	o-METHYLBENZOIC ACID - PFB	136	125	138	120	130	8.6	6.6
44	PHENYLACETIC ACID - PFB	139	133	137	127	134	5.3	4.0
45	m-TOLYLACETIC ACID - PFB	132	139	204	127	151	36.2	24.0
46	o-TOLYLACETIC ACID - PFB	144	144	156	125	142	12.8	9.0
47	2,6-DIMETHYLBENZOIC ACID - PFB	129	111	124	154	130	18.1	13.9
48	p-TOLYLACETIC ACID - PFB	138	134	217	136	156	40.6	25.9
49	p-METHYLBENZOIC ACID - PFB	140	137	165	129	143	15.5	10.8
50	3-PHENYLPROPANOIC ACID - PFB	136	135	129	131	133	3.3	2.5
51	2,5-DIMETHYLBENZOIC ACID - PFB	130	127	137	116	127	8.8	6.9
52	DECANOIC ACID - PFB	116	134	134	134	130	9.0	6.9
53	2,4-DIMETHYLBENZOIC ACID - PFB	131	127	137	122	130	6.3	4.9
54	3,5-DIMETHYLBENZOIC ACID - PFB	138	137	151	126	138	10.2	7.4
55	2,3-DIMETHYLBENZOIC ACID - PFB	138	132	139	128	134	5.5	4.1
56	4-ETHYLBENZOIC ACID - PFB	141	144	152	136	143	6.7	4.7
57	2,4,6-TRIMETHYLBENZOIC ACID - PFB	131	119	137	122	127	8.1	6.4
58	3,4-DIMETHYLBENZOIC ACID - PFB	144	138	164	133	145	13.5	9.3
59	2,4,5-TRIMETHYLBENZOIC ACID - PFB	125	125	126	119	124	3.3	2.7

Table IV. Quality Control for NICI/GC/MS Analysis for PFB Derivatives  
Phenols and Aliphatic and Aromatic Acids.

Sample	Frequency of Analysis	Quality Control Measure
50 ng DFTPP	Before each Calibration	EPA Tune Criteria must be met before Tuning for NICI.
NICI FC43 Tune	Before each Calibration	Intensity of ions 633, 414, 127 m/z are within $\pm 10\%$ of 100:44:12
Lab. Reagent Blank	Daily	Control chart of Int. Std. peak area vs time. Ensure that Int. Std. peak area is within $\pm 3 \times \text{S.D.}$ of average in control chart. Check for background contamination.
Check Standards at 10, 100 and 500 ppb	Daily	Ensure that calculated value obtained is within 10% of the expected value.
Lab. Fortified Blank Extraction (100 ml of Blank Water Spiked at 50 ppb)	Daily	Ensure that calculated value obtained is within 20% of the expected value.
Field Reagent Blank	As Provided	Check for background contamination.
Lab. Fortified Sample Matrix	As Required for Highly Contaminated Samples	Check for recovery of spiked compounds. Ensure that calculated value obtained is within 20% of the expected value.

# **Extraction of PCBs from Environmental Samples Using Accelerated Solvent Extraction (ASE)**

*Meets the requirements of U.S. EPA Method 3545 (Proposed)*

## **INTRODUCTION**

Accelerated Solvent Extraction (ASE™) is a new extraction method that significantly streamlines sample preparation. A commonly used solvent is pumped into an extraction cell containing the sample, which is then brought to an elevated temperature and pressure. Minutes later, the extract is transferred from the heated cell to a standard collection vial for cleanup or analysis. The entire extraction process is fully automated and performed in minutes for fast and easy extraction with low solvent consumption.

ASE is used as a direct replacement for solvent intensive techniques such as Soxhlet and sonication. For the preparation of solid waste samples containing PCBs, ASE provides more convenient, faster extractions with significantly less solvent usage than these other methods. ASE will extract a 10-g sample of a typical solid waste in about 10 min with a total solvent consumption of approximately 15 mL.

PCBs are found in many solid waste materials worldwide. This application note describes the application of ASE to the extraction of PCBs from sewage sludge, river sediments, marine sediments, and marine tissue (oyster). The procedures described in this application note meet the requirements for sample extraction as determined by U.S. EPA Method 3545 (Proposed) for solid samples.

## **EQUIPMENT**

Dionex ASE 200 Accelerated Solvent Extractor, with  
11-mL or larger stainless steel extraction cells  
GC with ECD  
Dionex Vials for collection of extracts  
(40 mL P/N 49465; 60 mL P/N 49466)

## **SOLVENTS**

Hexane  
Acetone

## **ASE 200 CONDITIONS**

System Pressure:	14 MPa (2000 psi)
Oven Temperature:	100 °C
Sample Size:	5 to 10 g
Oven Heat-up Time:	5 min
Static Time:	5 min
Flush Volume:	60% of extraction cell volume
Solvent:	Hexane/acetone (1:1), (v/v)
Nitrogen Purge:	1 MPa (150 psi) for 60 s

## **SAMPLE INFORMATION**

Sewage sludge was obtained from the Fresenius Institute (Taunusstein, Germany). Oyster tissue samples were obtained from the National Oceanographic and Atmospheric Administration (NOAA) Laboratory (Seattle, Washington, USA). The river sediment is a standard reference material, SRM 1939 (National Institute of Science and Technology, Gaithersburg, Maryland, USA). Contaminated soil used in this study was a certified reference material (CRM911-050) purchased from Resource Technology Corporation (Laramie, Wyoming, USA).

## **SAMPLE PREPARATION**

Samples should be dried and ground. Before filling the cell, a cellulose disk should be placed in the outlet end of the cell. Samples that contain water (greater than 10%) should be mixed in equal proportions with sodium sulfate or Hydromatrix™.

## **QUANTIFICATION OF SEWAGE SLUDGE, OYSTER TISSUE, AND RIVER SEDIMENT**

Sample extracts from ASE were prepared for analysis by passing through silver nitrate/sulfuric acid loaded silica gel and alumina columns followed by concentration to 1 mL for GC analysis. PCB analyses were performed by gas chromatography with ECD using a 30-m x 0.25-mm i.d., Rtx-5 (Restek, Bellefonte, Pennsylvania, USA) or equivalent column. Injector and detector were maintained at 300 °C. The GC oven was programmed from 100–300 °C at 10 °C/min following a 5-min hold. External standards were used for calibration.

## **QUANTIFICATION OF SOIL (CRM911-050)**

PCB analyses of the soil extracts were performed according to U.S. EPA SW-846 Method 8080. The ASE 200 extracts were diluted to 25 mL prior to analysis by GC. Injection was through a split/splitless injector in a GC with dual electron capture detectors. Two capillary columns, a 30-m x 0.53-mm i.d. DB-608 and a 30-m x 0.53-mm i.d. DB-1701 (J&W Scientific, Folsom, California, USA) provided primary and confirmation data, respectively. Both columns were joined with a fused silica "Y" connector (Restek). The remaining part of the "Y" was connected to a 5-m section of

deactivated 0.53-mm i.d. fused silica capillary tubing that acted as a guard column. The end of this guard column was inserted into the GC injector. Dual confirmation of the analytes was achieved with a single 5-μL injection. The injector was maintained at 220 °C and both detectors were operated at 320 °C. The oven was programmed from 60–200 °C at 28 °C/min after a 1-min hold, then 265 °C at 10 °C/min with a hold of 20.5 min. Helium was used as the carrier gas at a linear velocity of approximately 30 cm/s.

## **ANALYTICAL RESULTS**

Results from extractions of sewage sludge, oyster tissue, river sediment, and soil are shown in Tables 1 through 4. These tables show the average recoveries and RSDs (%) for PCB congener content of these matrices.<sup>1</sup> Recoveries for all compounds with the exception of one (PCB 153 from the river sediment) are above 77% of the certified or Soxhlet comparison values. Interferences in the river sediment extract also prevented quantification of two low molecular weight PCB congeners (PCB 28 and PCB 52).

The results demonstrate the effectiveness of ASE as a sample preparation method. ASE provides extracts with minimal solvent usage and significant time reduction compared to other extraction methods. Results are comparable to the traditional Soxhlet extraction method.

ASE meets the requirements for PCB analysis as described in U.S. EPA SW-846 Method 3545 (Proposed).

## **REFERENCES**

1. Richter, B.; Ezzell, J.; Felix, D. "Single Laboratory Method Validation Report: Extraction of Organophosphorous Pesticides, Chlorinated Herbicides, and Polychlorinated Biphenyls Using Accelerated Solvent Extraction (ASE) with Analytical Validation by GC/NPD and GC/ECD," Document 101124, Dionex Corporation, December 2, 1994.

**Table 1 PCB Recoveries from sewage sludge<sup>a</sup>**

PCB Congener	Average Recovery, n=6 (as % of Soxhlet)	RSD (%)
PCB 28	118.1	2.5
PCB 52	114.0	4.7
PCB 101	142.9	7.4
PCB 153	109.5	5.8
PCB 138	109.6	3.9
PCB 180	160.4	7.5

<sup>a</sup> Analyte concentration range: 160–200 µg/kg per component**Table 2 PCB Recovery from oyster tissue<sup>a</sup>**

PCB Congener	Average Recovery, n=6 (as % of Soxhlet)	RSD (%)
PCB 28	90.0	7.8
PCB 52	86.9	4.0
PCB 101	83.3	1.5
PCB 153	84.5	3.5
PCB 138	76.9	3.0
PCB 180	87.0	4.3

<sup>a</sup> Analyte concentration range: 50–150 µg/kg per component**Table 3 PCB Recovery from river sediment  
(SRM 1939)<sup>a</sup>**

PCB Congener	Average, n=6 (as % of Soxhlet)	RSD (%)
PCB 101	89.2	3.7
PCB 153	62.3	4.1
PCB 138	122.1	2.3
PCB 180	111.5	5.9

<sup>a</sup> Analyte concentration range: 170–800 µg/kg per component**Table 4 Recovery of Arochlor 1254 from soil  
(CRM911-050)**

Run Number	Arochlor Found (µg/kg)
1	1290.0
2	1365.8
3	1283.4
4	1368.6
Average	1327.0 (99.0%)
RSD	3.51%

APPENDIX B  
R.S. Kerr Laboratory Data

Results from First Sampling Event  
(Day 0)

Sampling Date: 15-July-1998

R.S. Kerr Laboratory Service Request Number: SF-0-1

Table I. Quantitative Report and QC Data for Polychlorinated Biphenyls  
In Core Samples from Cape Canaveral Service Request SF-0-1.

Concentration  $\mu\text{g PCB/g dry soil}$

File Name:	352A11.TXT	353A12.TXT	354A13.TXT	355A14.TXT	356A15.TXT	357A16.TXT	359A21.TXT	360A22.TXT	361A23.TXT
PCB Congener	A1-1	A1-2	A1-3	A1-4	A1-5	A1-6	A2-1	A2-2	A2-3
26-4	0.96	10.89	0.79	0.89	0.90	0.92	1.01	0.98	1.05
25-26	1.77	***	0.79	1.54	1.28	1.20	2.33	2.18	2.75
24-4	3.09	2.69	1.83	2.62	2.57	2.46	4.43	3.69	4.93
24-26	0.41	0.85	0.38	0.51	0.48	0.39	0.83	.	0.87
236-2	0.81	6.88	0.75	0.93	0.86	0.96	1.05	1.11	1.27
23-4	0.30	0.52	0.46	0.84	0.33	0.77	1.71	1.47	0.83
23-26	0.66	5.69	0.54	0.70	0.62	0.87	0.75	0.76	0.83
25-25	9.03	8.86	5.13	8.55	6.71	7.76	12.72	11.88	13.66
24-25	6.59	6.46	4.21	6.17	5.42	5.93	10.14	9.17	10.26
24-24	3.09	3.02	1.63	2.57	2.28	2.60	4.17	3.69	4.36
23-25	10.55	10.89	6.42	10.00	8.04	6.26	16.19	15.48	16.93
236-3	0.81	7.35	0.71	0.89	0.81	0.87	0.70	1.29	1.05
23-24	4.41	0.85	1.88	3.18	2.90	3.42	6.45	4.63	6.28
26-34	3.04	1.65	1.33	2.66	2.28	2.41	4.48	4.18	4.76
234-2	0.76	6.83	0.50	0.65	0.71	0.58	0.79	0.67	0.79
236-4	5.88	5.84	3.67	5.28	4.66	5.20	8.95	7.70	8.29
23-23	3.96	2.92	2.96	3.55	3.47	3.04	5.05	3.16	5.02
235-4	0.96	7.31	0.67	0.79	0.81	0.87	1.10	0.80	1.22
236-25	1.52	7.21	1.13	1.45	1.33	1.45	1.97	1.96	2.05
245-4	4.97	4.95	2.92	4.81	3.90	4.05	7.90	7.47	8.55
25-34	1.32	7.21	0.92	0.93	1.05	1.01	1.45	1.69	1.40
24-34	9.79	9.38	6.17	9.02	7.90	8.09	15.40	14.77	15.67
235-25	1.17	0.28	0.92	1.12	1.05	1.01	0.97	1.33	0.92
23-34	4.21	5.47	3.42	5.37	4.47	2.12	8.78	8.19	8.90
235-24 or 245-25	4.77	4.24	2.83	4.25	3.90	4.05	6.80	5.47	7.07
234-4	1.62	7.40	1.13	1.17	1.47	1.45	1.93	2.09	2.09
245-24	2.18	0.66	1.21	1.31	1.62	1.83	3.60	3.07	3.97
235-23	2.54	2.40	1.54	2.43	2.19	2.46	2.24	3.69	3.97
234-25	2.33	1.98	1.33	1.49	1.33	1.93	2.76	3.69	3.97
234-24	2.33	3.02	1.46	2.15	1.62	1.06	2.90	2.09	1.27
236-34	5.07	4.57	3.21	4.53	4.09	4.63	7.72	7.79	8.12
234-23	0.30	0.75	0.46	1.03	0.48	0.39	0.48	1.69	1.83
34-34	0.81	1.27	0.75	0.89	0.90	0.96	1.54	1.47	1.13
245-34	4.82	4.76	3.38	4.44	4.00	4.38	6.05	6.10	6.28
234-34	3.55	2.59	2.58	3.60	2.66	3.37	4.91	5.25	5.72
Total PCB	110.39	158.65	69.96	102.31	89.09	90.54	160.24	150.65	168.07

\*\*\* indicates concentration of extract was below lowest calibration standard (0.1 ppm)

. indicates not found.

Table I. Quantitative Report and QC Data for Polychlorinated Biphenyls  
In Core Samples from Cape Canaveral Service Request SF-0-1.

Concentration  $\mu\text{g PCB/g dry soil}$

File Name:	362A24.TXT	363A25.TXT	364A25D.TXT	366A27.TXT	367X11.TXT	368X12.TXT	369X13.TXT	370X14.TXT	371X15.TXT
PCB Congener	A2-4	A2-5	A2-6	A2-7	X1-1	X1-2	X1-3	X1-4	X1-5
26-4	1.16	0.98	0.96	0.91	0.86	0.77	0.80	***	0.77
25-26	3.08	2.86	1.58	2.13	***	0.36	0.40	0.32	0.36
24-4	5.62	6.04	3.36	4.21	0.34	0.41	0.35	0.32	0.31
24-26	0.67	0.94	*	*	***	***	*	***	***
236-2	1.43	1.39	0.91	1.22	0.74	0.67	0.65	0.63	0.67
23-4	1.74	2.15	1.15	1.57	***	***	***	***	***
23-26	0.85	0.85	0.67	0.81	***	***	0.60	***	***
25-25	15.96	15.30	8.78	11.76	1.43	1.44	1.20	1.37	1.44
24-25	11.90	8.41	6.53	9.02	1.77	1.54	1.55	1.64	1.64
24-24	4.86	4.88	3.02	3.95	0.92	0.98	1.05	0.90	0.92
23-25	19.39	19.23	11.47	14.60	1.60	1.59	1.85	1.74	1.39
236-3	*	1.48	0.72	1.06	0.74	0.67	0.70	***	0.67
23-24	7.93	6.75	5.14	5.78	2.00	1.90	1.90	1.96	1.85
26-34	5.30	5.37	3.22	4.21	0.46	0.46	0.30	0.42	0.51
234-2	0.85	0.85	0.62	0.81	*	***	***	***	***
236-4	9.76	11.23	6.48	8.06	1.03	1.44	1.30	1.22	1.44
23-23	5.44	5.14	4.03	4.26	***	***	2.35	***	*
235-4	1.16	1.34	0.82	1.17	0.80	***	***	0.74	***
236-25	2.41	1.74	1.63	2.03	0.92	0.82	0.80	0.90	0.87
245-4	9.27	8.99	5.81	7.20	0.69	0.46	0.75	0.85	0.41
25-34	1.38	1.61	1.25	1.11	0.86	0.77	0.75	0.79	0.82
24-34	17.65	18.03	11.19	9.38	1.26	1.85	1.10	1.90	1.85
235-25	0.98	1.57	1.06	1.47	***	***	***	***	***
23-34	10.07	10.20	6.77	8.56	0.69	0.82	1.00	0.79	0.51
235-24 or 245-25	8.25	7.42	5.23	6.64	1.72	1.65	1.50	1.48	1.39
234-4	2.32	2.19	1.82	2.13	0.92	0.82	0.90	0.85	0.82
245-24	4.23	4.16	2.54	3.14	0.17	0.15	0.15	0.00	0.10
235-23	4.68	4.34	2.26	3.65	1.14	0.93	0.95	1.06	0.98
234-25	4.10	4.12	1.97	2.53	0.34	0.46	0.45	0.42	0.51
234-24	1.74	2.77	2.59	2.48	***	1.03	***	***	***
236-34	9.45	8.90	6.38	7.55	1.14	0.87	0.90	0.79	0.82
234-23	2.23	*	1.20	1.72	0.29	0.26	***	0.21	***
34-34	1.83	1.57	0.91	*	0.63	0.51	***	0.58	0.56
245-34	*	7.42	*	*	1.72	1.85	1.85	1.90	1.85
234-34	6.60	4.07	4.37	5.27	1.55	1.54	1.30	1.37	1.54
Total PCB	184.24	184.28	116.46	140.38	26.73	27.06	27.40	25.15	25.01

\*\*\* indicates concentration of extract was below lowest calibration standard (0.1 ppm)

\* indicates not found.

Table I. Quantitative Report and QC Data for Polychlorinated Biphenyls  
In Core Samples from Cape Canaveral Service Request SF-0-1.

Concentration  $\mu\text{g PCB/g dry soil}$

File Name:	372X17.TXT	373X21.TXT	374X22.TXT	375X23.TXT	376X24.TXT	377X25.TXT	378X26.TXT	379X27.TXT	380N11.TXT
PCB Congener	X1-7	X2-1	X2-2	X2-3	X2-4	X2-5	X2-6	X2-7	N1-1
26-4	***	0.92	0.82	***	0.89	***	0.75	0.88	0.80
25-26	0.38	0.40	0.36	0.40	***	0.39	0.30	0.39	0.35
24-4	0.27	0.34	0.41	0.20	0.42	0.33	0.25	0.55	0.85
24-26	***	***	***	***	*	***	***	***	0.40
236-2	0.66	0.75	0.67	0.65	0.77	0.72	***	0.72	0.70
23-4	***	***	***	***	***	***	***	***	***
23-26	***	***	***	0.60	***	***	***	***	0.60
25-25	1.42	1.55	1.24	1.36	1.42	1.33	1.40	2.65	2.80
24-25	1.64	1.90	1.65	1.96	2.02	1.72	1.64	2.21	2.40
24-24	0.87	1.03	0.93	1.11	1.19	0.89	0.90	1.16	0.80
23-25	1.70	1.32	2.32	2.01	1.78	1.55	1.59	2.38	2.90
236-3	***	***	0.67	0.65	0.77	0.72	***	***	0.70
23-24	1.75	2.13	2.01	1.91	2.02	1.88	1.89	2.10	2.15
26-34	0.33	0.40	0.57	0.65	0.42	0.50	0.40	0.61	0.90
234-2	0.66	***	0.62	***	***	***	***	0.66	***
236-4	0.82	1.26	1.60	1.61	1.66	1.44	1.49	1.88	2.15
23-23	*	***	***	2.46	***	***	***	***	*
235-4	***	***	0.72	***	***	***	0.70	0.77	0.70
236-25	0.87	0.98	0.82	0.85	1.01	0.89	0.80	1.00	0.85
245-4	0.55	1.03	1.13	0.85	0.89	0.50	0.80	1.22	1.45
25-34	0.77	0.80	0.82	0.80	0.89	0.83	0.75	0.88	0.95
24-34	1.80	2.24	1.65	2.62	2.19	1.88	1.84	1.44	3.25
235-25	***	*	***	0.96	***	***	***	***	***
23-34	0.82	0.86	1.13	1.16	1.01	0.77	0.40	0.88	1.60
235-24 or 245-25	1.64	1.49	1.60	2.01	1.84	1.33	1.49	1.22	1.60
234-4	0.93	0.98	0.93	0.85	1.01	0.89	0.80	0.94	0.95
245-24	0.22	0.17	0.41	0.35	0.30	0.11	0.25	0.44	0.60
235-23	1.20	1.09	1.18	1.11	1.25	1.22	1.00	1.22	1.40
234-25	0.49	0.46	0.41	0.60	0.36	0.33	0.40	0.72	0.70
234-24	***	***	***	***	***	***	***	1.11	***
236-34	1.15	1.26	1.18	1.61	0.95	0.83	1.15	1.44	1.75
234-23	***	***	0.26	*	0.30	***	0.25	0.28	0.35
34-34	***	*	0.57	0.55	0.65	***	***	0.61	0.55
245-34	1.80	2.47	2.26	2.46	2.08	2.27	1.99	2.10	2.65
234-34	1.42	1.44	1.34	1.66	1.72	1.61	1.30	1.88	1.95
Total PCB	24.17	27.31	30.26	34.05	29.77	24.91	24.51	33.73	39.75

\*\*\* indicates concentration of extract was below lowest calibration standard (0.1 ppm)

\* indicates not found.

Table I. Quantitative Report and QC Data for Polychlorinated Biphenyls  
In Core Samples from Cape Canaveral Service Request SF-0-1.

Concentration  $\mu\text{g PCB/g dry soil}$

File Name:	381N12.TXT	382N13.TXT	383N14.TXT	384N15.TXT	385N16.TXT	386N17.TXT	387N21.TXT	388N22.TXT	389N23.TXT
PCB Congener	N1-2	N1-3	N1-4	N1-5	N1-6	N1-7	N2-1	N2-2	N2-3
26-4	0.84	0.86	0.62	0.80	0.61	0.71	0.75	0.83	0.67
25-26	1.39	0.59	0.59	0.55	0.57	0.66	0.55	0.70	0.55
24-4	2.74	1.19	1.14	0.85	0.57	0.93	0.89	0.57	0.67
24-26	0.55	***	0.29	***	0.36	***	***	***	***
236-2	0.84	0.76	0.55	0.70	0.53	0.62	0.70	0.67	0.59
23-4	0.59	0.16	0.22	0.00	0.00	0.13	***	***	0.08
23-26	0.59	0.65	0.48	0.60	0.43	***	0.61	***	***
25-25	7.33	2.05	3.57	3.14	3.10	3.13	3.71	2.12	3.07
24-25	5.06	2.97	2.90	2.74	2.71	2.69	2.82	2.23	2.65
24-24	2.53	1.57	1.10	1.15	0.75	1.37	1.55	1.04	1.30
23-25	8.21	4.05	3.75	2.04	3.35	3.70	4.04	2.59	2.31
236-3	0.72	0.70	0.59	0.75	0.53	0.82	0.70	0.67	0.63
23-24	3.50	2.11	1.87	2.24	1.64	2.16	2.39	2.07	2.23
26-34	2.32	1.19	1.10	1.00	0.82	1.06	1.13	0.78	1.01
234-2	0.63	0.65	0.48	***	0.43	0.53	0.56	***	0.55
236-4	4.59	2.65	2.43	1.74	2.17	2.25	2.44	1.29	2.44
23-23	3.20	2.97	2.17	2.69	2.07	2.34	***	***	2.48
235-4	0.76	0.81	0.55	0.75	0.57	0.66	0.75	0.73	***
236-25	1.31	1.13	0.74	0.95	0.75	1.01	1.03	0.93	0.92
245-4	4.17	2.05	1.87	1.79	1.43	1.01	2.11	1.35	1.85
25-34	1.10	1.08	0.81	1.00	0.75	0.88	0.80	0.83	0.84
24-34	7.96	4.48	3.90	3.84	3.85	2.07	4.22	2.69	3.82
235-25	1.18	***	0.77	0.95	0.71	0.88	0.99	***	***
23-34	4.51	1.84	2.10	1.15	1.85	2.20	2.21	1.45	1.64
235-24 or 245-25	4.21	2.59	2.35	1.49	2.17	2.29	2.02	2.07	1.47
234-4	1.31	1.13	0.88	1.05	0.71	0.97	0.99	0.98	0.97
245-24	1.10	0.76	0.85	0.50	0.36	0.53	0.94	0.26	0.88
235-23	1.56	1.62	1.40	1.49	1.25	1.45	1.41	1.40	1.47
234-25	1.52	0.59	0.96	0.80	0.89	0.79	1.13	0.73	0.92
234-24	1.52	1.30	1.07	1.15	0.93	1.15	1.13	***	1.09
236-34	3.92	1.08	1.69	2.04	1.11	1.50	2.35	1.71	2.23
234-23	1.05	0.49	0.44	0.35	0.36	0.44	0.38	0.36	0.38
34-34	1.05	0.70	0.62	0.75	0.50	0.71	*	0.62	0.67
245-34	*	3.19	2.57	3.04	2.50	2.73	2.96	2.59	*
234-34	3.16	1.89	1.40	2.24	1.82	1.59	2.16	1.50	1.97
Total PCB	86.99	51.84	48.82	46.09	43.14	45.74	50.54	35.42	42.33

\*\*\* indicates concentration of extract was below lowest calibration standard (0.1 ppm)

\* indicates not found.

Table I. Quantitative Report and QC Data for Polychlorinated Biphenyls  
In Core Samples from Cape Canaveral Service Request SF-0-1.

Concentration  $\mu\text{g PCB/g dry soil}$

File Name:	380N24.TXT	391N25.TXT	392N26.TXT	393N26.TXT	394N15D.TXT
PCB Congener	N2-4	N2-5	N2-6	N2-6 dup	A1-5 dup
26-4	0.78	0.54	0.55	0.40	0.60
25-26	0.95	0.59	0.69	0.52	0.65
24-4	1.86	1.40	1.57	1.09	1.59
24-26	0.34	0.27	0.32	0.20	0.30
236-2	1.05	0.68	0.78	0.60	0.80
23-4	0.85	0.63	0.69	0.49	0.70
23-26	0.61	0.54	0.60	0.43	0.55
25-25	4.94	3.17	3.82	2.96	3.44
24-25	3.72	2.27	2.63	2.04	2.39
24-24	1.56	1.04	1.24	0.92	1.20
23-25	5.27	3.58	4.33	3.25	3.74
236-3	0.34	0.41	0.46	0.32	0.40
23-24	1.96	1.31	1.57	1.26	1.54
26-34	1.76	1.31	1.43	1.09	1.35
234-2	0.68	0.50	0.64	0.43	0.60
236-4	2.84	1.81	2.17	1.67	1.89
23-23	1.42	1.27	1.34	1.01	1.30
235-4	0.44	0.41	0.41	0.29	0.45
236-25	2.03	1.54	1.70	1.38	1.54
245-4	2.74	2.04	2.49	1.95	2.19
25-34	1.62	1.27	1.52	1.12	1.59
24-34	5.41	3.85	4.79	3.85	4.14
235-25	***	***	***	***	***
23-34	3.01	2.31	2.76	2.13	2.49
235-24 or 245-25	2.37	1.81	2.07	1.69	1.84
234-4	1.96	1.45	1.89	1.44	1.69
245-24	1.62	1.36	1.52	1.21	1.44
235-23	1.25	1.00	1.15	0.92	1.10
234-25	1.69	1.36	1.57	1.21	1.44
234-24	1.22	1.18	1.29	1.01	1.20
236-34	3.18	2.45	2.90	2.38	2.64
234-23	1.15	1.04	1.20	0.89	1.15
34-34	0.85	0.82	0.92	0.66	0.85
245-34	2.50	2.04	2.53	1.87	2.34
234-34	2.27	2.13	2.44	1.72	2.34
Total PCB	66.21	49.39	58.00	44.38	53.46

\*\*\* indicates concentration of extract was below lowest calibration standard (0.1 ppm)

. indicates not found.

25

Cape Canaveral Fla.

7-25-98

2

# MOISTURE

and

### Preparation

SF-0-1

Feb

ASE

**SECRET**

(ACB)

Sample #	Initial wt. WT. / DRY	(Boehm) Final wt. WT. / DRY	(Baker) Tare wt. WT. / DRY	% Moisture
① A1-1	9.96 8.50	39.79	30.99	14.83
② A1-2 removed approx 1.5ml	9.86 8.72	38.95	30.23	11.56
③ A1-3	15.87 9.96	40.70	30.80	37.24
④ A1-4	9.95 8.44	39.62	30.03	36.5
⑤ A1-5	9.90 8.57	40.35	31.52	10.61
⑥ A1-6	10.30 8.53	39.30	30.77	17.18
⑦ A1-7	9.40 9.19	38.85	32.22	2.23
⑧ A2-1	10.50 9.39	37.86	28.47	10.57
⑨ A2-2	10.30 9.08	39.55	30.47	11.84
⑩ A2-3	10.60 9.44	37.89	28.45	10.94
⑪ A2-4	10.31 9.02	40.27	31.25	12.51
⑫ A2-5	10.40 9.30	56.53	47.14	12.24
⑬ A2-6 duplicate	9.25 8.02	57.88	49.86	13.29
⑭ A2-6 "	9.94 8.47	58.46	49.99	14.78
⑮ A2-7	9.75 8.09	58.64	50.55	17.02
⑯ X1-1	9.97 7.11	55.81	48.70	28.69
⑰ X1-2	10.09 7.97	58.30	50.33	21.01
⑱ X1-3	9.66 8.24	58.44	50.20	14.69
⑲ X1-4	10.30 7.91	57.97	50.06	23.20
⑳ X1-5	9.52 8.16	69.14	60.98	14.28
㉑ X1-6	9.95 8.68	53.15	48.38	12.76
㉒ Methoc Blank				

8-3-98

SF-1

ASE

Cape Canaveral

% Moisture and PCB extraction

8398

50-1

Sample #	Soil sample		Wet Beaker		Beaker	% moisture	Samples sample #
	Wet weight	Dry wt.	Final wt.	Initial wt.			
X1-7	10.05	8.23	40.60	38.70	30.55	18.10	X1-1
X2-1	10.11	7.41	39.31	36.61	29.19	26.70	X1-7
X2-2	10.07	8.51	39.75	38.19	29.68	15.49	X1-6
X2-3	10.30	8.53	39.84	39.27	29.54	17.18	X1-6
X2-4	10.33	7.25	41.84	38.76	31.51	29.81	X1-6
X2-5	7.86	7.75	39.77	38.61	29.92	2.639	X1-6
X2-6	7.94	8.49	40.74	39.49	30.00	14.58	
X2-7	7.47	7.74	39.65	37.30	28.44	18.26	
N1-1	10.04	8.57	40.83	39.36	30.79	14.64	
N1-2	10.67	9.78	39.77	38.25	28.47	8.34	
N1-3	10.24	7.87	40.50	38.13	30.26	23.14	
N1-4	15.09	11.48	44.35	40.74	29.26	23.92	
N1-5	10.10	8.43	39.81	38.14	29.71	16.53	
N1-6	10.56	12.06	42.35	38.45	26.79	22.49	
N1-7	10.55	9.19	39.86	38.50	29.31	12.89	
N2-1	10.29	8.95	40.24	38.90	29.65	13.02	
N2-2	7.95	8.11	39.24	37.40	29.29	18.49	
N2-3	10.57	9.93	40.09	39.45	29.52	6.05	
N2-4	10.70	12.51	45.38	42.19	29.68	20.31	
N2-5	10.31	9.38	38.84	37.91	28.53	9.02	
N2-6	9.78	9.16	40.45	39.63	30.47	8.21	
N2-6 2	10.61	9.40	41.86	40.65	31.25	11.40	
N1-5 AP	10.91	8.43	40.03	38.32	29.89	16.86	
Method						0.0	
Blank						0.0	
PCB ST	1.001					KPS	
						MAR	

MANTECH ENVIRONMENTAL RESEARCH SERVICES CORPORATION

PEAK RESULTS -QC SAMPLES

#	SampleName	acetic acid(uM)	butyric acid (uM)
1	HPLC BLANK	ND	ND
2	CS 15 uM	15.9	15.7
3	STD 25 uM	25.7	25.5
4	HPLC BLANK	ND	ND
5	CS 15 uM	16.2	15.2
6	STD 25 uM	25.0	25.9

SampleName acetic acid (mg/kg) butyric acid (mg/kg)

X1 4	9.25	ND
N1 4	7.83	ND
N2 4	10.9	ND
A1 4	30.0	114
A1 7	59.2	194
A2 4	21.9	87.7
A2 7	105	237
X2 4	10.9	ND

August 19, 1998

Don Kampbell's Cape Canaveral, FL  
Soil Extracts  
SF-0-1

Page 1

Sample	pH	Chloride mg/Kg soil	Sulfate mg/Kg soil
A1-2	8.98	14.5	48.5
A1-5	8.71	15.5	69.5
A2-2	9.10	9.66	30.8
A2-5	8.90	10.7	38.2
N1-2	8.32	10.2	12.7
N1-5	8.35	10.8	9.51
N1-5 dup	—	10.8	9.32
N2-2	8.31	15.5	11.7
N2-5	8.29	13.8	14.4
X1-2	8.08	1530	1280
X1-5	8.07	1580	1170
X2-2	8.02	1880	1440
X2-5	8.01	2060	1540
X2-5 dup	8.01	—	—
Blank	—	<5	<5
WPO39	—	10.8	57.1
WPO39 T.V.	—	10.8	58.0
Spike Recovery	—	98%	95%
Sample	Nitrate mg/Kg soil		
A1-1	<1		
A1-6	<1		
A2-1	<1		
A2-6	<1		
N1-1	<1		
N1-6	<1		
N2-1	<1		
N2-1 dup	<1		
N2-6 (1)	<1		
N2-6 (2)	<1		
X1-1	29.4		
X1-6	84.1		
X2-1	77.8		
X2-6	67.4		
X2-6 dup	67.3		
Blank	<1		
WPO39	1.07		
WPO39 T.V.	1.10		
Spike Recovery	102%		

Results from Second Sampling Event  
(Day 42)

Sampling Date: 26-August-1998

R.S. Kerr Laboratory Service Request Number: SF-0-15

Table I. Quantitative Report and QC Data for Polychlorinated Biphenyls in Core Samples  
from Cape Canaveral (Second Sample Set) Service Request SF-0-15.

Concentration  $\mu\text{g PCB/g dry soil}$

File Name:	423A11.TXT	424A12.TXT	425A13.TXT	426A14.TXT	427A15.TXT	428A16.TXT	428A17.TXT	431A21.TXT	432A22.TXT
PCB Congener	A1-1	A1-2	A1-3	A1-4	A1-5	A1-6	A1-7	A2-1	A2-2
26-4	0.57	0.83	0.68	0.58	0.49	0.66	0.51	1.47	1.64
25-26	1.31	1.73	1.44	1.28	1.20	1.40	1.05	2.72	2.86
24-4	0.94	1.18	1.00	0.89	0.90	0.85	0.82	1.17	1.17
24-26	0.37	0.59	0.52	0.47	0.41	0.43	0.43	0.87	0.82
236-2	1.52	2.08	1.88	1.51	1.50	1.59	1.44	2.76	3.01
23-4	0.90	1.26	1.04	0.85	0.86	0.97	0.86	1.59	1.60
23-26	0.82	1.06	0.92	0.81	0.82	0.89	0.78	1.40	1.49
25-25	8.04	10.78	8.80	7.99	7.53	8.47	7.43	14.95	14.17
24-25	5.74	8.06	6.16	5.54	5.58	6.18	5.29	10.72	10.68
24-24	2.22	3.19	2.52	2.40	2.17	2.37	2.02	4.12	3.95
23-25	8.78	12.27	9.88	9.38	8.54	9.59	8.21	17.18	16.71
236-3	0.57	0.98	0.64	0.66	0.52	0.66	0.58	1.32	1.06
23-24	3.24	4.01	3.52	2.79	2.73	2.99	3.07	5.55	5.09
26-34	2.01	3.07	2.16	2.17	1.87	2.25	1.83	4.72	4.81
234-2	0.78	0.94	0.76	0.74	0.75	0.78	0.82	1.40	1.37
236-4	4.51	6.49	4.96	4.77	4.38	5.17	4.24	9.06	8.69
23-23	2.13	2.32	2.36	2.05	2.06	1.94	1.98	3.78	3.48
235-4	0.53	0.71	0.60	0.58	0.52	0.54	0.51	0.94	0.94
236-25	2.87	3.85	3.00	3.06	2.73	3.11	2.72	5.17	5.20
245-4	3.73	5.27	4.04	3.99	3.67	4.27	3.58	7.21	7.16
25-34	0.90	1.26	0.96	1.01	0.90	0.89	0.86	1.59	1.45
24-34	7.47	10.97	8.16	8.22	7.56	8.31	7.19	14.77	14.48
235-25	1.03	1.18	1.16	1.01	0.94	1.20	1.01	1.51	1.45
23-34	4.47	6.29	4.80	4.81	4.38	5.28	4.47	8.95	8.57
235-24 or 245-25	3.16	4.48	3.40	3.26	3.15	3.65	3.03	6.16	5.95
234-4	2.91	4.09	2.88	3.14	2.73	3.30	2.61	5.29	5.09
245-24	2.26	3.15	2.44	2.36	2.17	2.41	2.10	4.15	4.03
235-23	1.68	2.40	1.84	1.86	1.69	1.98	1.59	3.32	3.29
234-25	2.26	2.99	2.32	2.21	2.13	2.64	2.22	4.15	4.19
234-24	1.76	1.34	1.72	1.59	1.65	2.10	1.71	2.98	2.74
236-34	4.51	6.17	4.52	4.81	4.12	4.93	4.32	8.35	8.14
234-23	1.48	2.20	1.60	1.59	1.31	1.79	1.52	2.79	2.66
34-34	1.07	1.22	1.08	1.05	1.01	1.17	1.01	1.66	1.60
245-34	3.16	4.44	3.40	3.53	3.03	3.77	3.19	5.97	5.91
234-34	2.83	3.78	2.84	2.98	2.77	3.15	2.84	5.06	4.74
Total PCB	92.55	126.63	99.98	95.94	88.79	101.67	87.62	174.80	170.20

\*\*\* indicates concentration of extract was below lowest calibration standard (0.1 ppm)

• indicates not found.

Table I. Quantitative Report and QC Data for Polychlorinated Biphenyls in Core Samples  
from Cape Canaveral (Second Sample Set) Service Request SF-0-15.

Concentration µg PCB/g dry soil												
433A23.TXT	434A24.TXT	435A25.TXT	436A26.TXT	437A27.TXT	439X11.TXT	440X12.TXT	441X13.TXT	442X14.TXT				
A2-3	A2-4	A2-5	A2-6	A2-7	X1-1	X1-2	X1-3	X1-4				
2.07	1.52	1.77	1.72	1.57	0.40	0.39	0.34	0.39				
3.25	2.69	2.95	2.84	2.87	0.48	0.46	0.41	0.50				
1.40	1.10	1.51	1.16	1.49	0.95	1.03	0.94	1.04				
1.03	0.81	0.81	0.93	0.84	0.18	0.18	0.19	0.18				
3.66	2.76	3.17	2.99	3.14	0.55	0.57	0.49	0.54				
1.92	1.52	1.73	1.68	1.64	0.44	0.43	0.41	0.47				
1.74	1.45	1.03	1.42	1.26	0.40	0.39	0.38	0.43				
17.73	14.52	16.22	14.99	15.72	2.30	2.53	2.11	2.54				
13.22	10.80	12.13	11.51	11.47	1.43	1.74	1.43	1.68				
5.13	3.93	4.20	4.07	4.40	0.73	0.78	0.72	0.82				
20.20	16.78	18.21	17.53	17.63	2.41	2.71	2.33	2.65				
0.85	0.74	1.03	1.42	1.11	0.29	0.25	0.30	0.29				
6.98	4.32	6.38	5.05	6.58	0.95	1.07	0.90	1.04				
5.69	4.74	5.23	5.08	4.90	0.95	1.03	0.90	0.97				
1.44	1.31	1.33	1.23	1.49	0.33	0.39	0.34	0.43				
10.19	9.06	9.10	8.63	7.80	1.21	1.39	1.17	1.33				
4.25	3.79	3.58	3.48	3.56	0.84	0.93	0.90	0.97				
1.11	0.60	0.55	0.56	0.80	0.26	0.25	0.26	0.29				
5.95	5.38	5.45	5.46	5.55	1.02	1.21	1.02	1.11				
9.05	7.44	8.00	7.14	7.42	1.32	1.57	1.32	1.54				
1.77	1.49	1.73	1.57	1.26	0.84	1.07	0.90	0.90				
16.80	14.62	15.26	15.06	15.22	2.41	3.03	2.48	2.87				
1.74	1.10	0.81	1.42	1.22	0.77	0.75	0.75	0.68				
10.27	8.67	9.25	8.93	8.87	1.54	1.82	1.62	1.72				
6.83	6.02	6.01	6.13	6.35	1.17	1.50	1.20	1.33				
6.17	5.17	3.13	5.38	5.51	1.06	1.21	1.05	1.11				
4.47	4.32	4.20	3.66	4.51	0.91	1.03	0.94	0.97				
3.73	3.36	3.43	3.59	3.56	0.62	0.78	0.68	0.75				
4.58	4.14	4.24	4.15	4.13	0.88	1.03	0.90	0.90				
2.55	2.66	2.73	3.18	2.91	0.77	0.89	0.75	0.86				
9.01	5.84	8.37	8.71	8.53	1.61	1.82	1.62	1.72				
2.95	2.73	2.06	2.50	2.33	0.77	0.82	0.75	0.79				
1.62	1.17	1.58	1.53	1.76	0.59	0.64	0.64	0.61				
5.87	5.63	5.93	4.90	5.35	1.32	1.60	1.43	1.43				
5.06	4.78	4.94	4.90	5.05	1.50	1.64	1.47	1.47				
200.27	166.98	178.06	174.53	177.82	34.21	38.92	34.07	37.30				

\*\*\* indicates concentration of extract was below lowest calibration standard (0.1 ppm)  
 . indicates not found.

Table I. Quantitative Report and QC Data for Polychlorinated Biphenyls in Core Samples  
from Cape Canaveral (Second Sample Set) Service Request SF-0-15.

Concentration  $\mu\text{g PCB/g dry soil}$

440X15.TXT	440X16.TXT	445X17.TXT	447X21.TXT	448X22.TXT	449X23.TXT	450X24.TXT	451X25.TXT	452X26.TXT
X1-5	X1-6	X1-7	X2-1	X2-2	X2-3	X2-4	X2-5	X2-6
0.37	0.35	0.33	0.33	0.35	0.33	0.37	0.38	0.38
0.37	0.35	0.43	0.40	0.39	0.46	0.37	0.45	0.50
0.92	1.01	0.93	0.88	0.92	0.69	1.05	0.94	1.04
0.17	0.17	0.17	0.18	0.21	0.13	0.19	0.19	0.19
0.51	0.45	0.46	0.44	0.56	0.46	0.45	0.45	0.46
0.34	0.49	0.33	0.37	0.49	0.33	0.49	0.45	0.50
0.37	0.38	0.36	0.40	0.39	0.31	0.41	0.41	0.46
2.24	2.41	2.22	2.09	2.61	2.24	2.36	2.11	2.57
1.53	1.64	1.53	1.47	1.80	1.65	1.65	1.47	1.84
0.58	0.63	0.73	0.73	0.67	0.76	0.75	0.64	0.85
2.48	2.76	2.49	2.31	2.15	2.62	2.51	2.41	2.15
0.24	0.21	0.20	0.22	0.74	0.18	0.71	0.60	0.27
0.78	0.91	0.86	0.70	1.09	1.02	1.05	0.90	0.85
0.92	0.91	0.86	0.88	0.95	0.53	0.97	0.75	1.00
0.31	0.35	0.33	0.37	0.39	0.20	0.37	0.34	0.42
1.19	1.08	1.09	1.14	1.34	1.37	1.31	1.20	1.46
0.78	0.84	0.76	0.73	0.85	0.81	0.97	0.90	0.88
0.20	0.24	0.23	0.22	0.28	0.23	0.26	0.26	0.27
1.05	1.08	1.06	0.84	1.20	1.02	1.12	0.83	1.00
1.29	1.43	1.33	0.99	1.45	1.45	1.35	0.90	1.61
0.65	0.91	0.83	0.88	0.92	0.87	0.94	0.90	1.00
2.55	2.55	2.55	2.50	3.07	2.70	2.85	2.45	3.00
0.71	0.70	0.63	0.73	0.74	0.56	0.79	0.75	0.81
1.56	1.61	1.49	1.06	1.73	1.65	1.69	1.51	1.81
0.85	1.26	1.23	1.06	1.06	1.20	1.27	1.17	1.38
0.98	1.05	0.76	0.73	1.13	1.02	1.12	1.02	1.15
0.82	0.91	0.90	0.88	0.81	0.92	0.94	0.90	1.04
0.68	0.73	0.56	0.70	0.74	0.46	0.64	0.64	0.73
0.88	0.70	0.93	0.84	0.71	0.89	0.94	0.90	1.00
0.75	0.63	0.76	0.77	0.81	0.51	0.52	0.79	0.88
1.60	1.71	1.63	1.73	1.80	1.68	1.72	1.69	1.81
0.71	0.63	0.73	0.73	0.56	0.38	0.75	0.72	0.77
0.54	0.59	0.56	0.55	0.46	0.33	0.67	0.64	0.54
1.29	1.29	1.33	1.43	1.38	1.42	1.35	1.32	1.15
1.39	1.29	1.49	1.58	1.62	1.12	1.54	1.54	1.69
32.60	34.30	33.11	31.91	36.38	32.49	36.48	33.55	37.45

\*\*\* Indicates concentration of extract was below lowest calibration standard (0.1 ppm)

\* Indicates not found.

Table I. Quantitative Report and QC Data for Polychlorinated Biphenyls in Core Samples  
from Cape Canaveral (Second Sample Set) Service Request SF-0-15.

Concentration µg PCB/g dry soil

File Name:	471X26D.TXT	453X27.TXT	455N11.TXT	455N12.TXT	457N13.TXT	458N14.TXT	458N15.TXT	460N16.TXT	461N17.TXT
PCB Congener	X2-6 dup	X2-7	N1-1	N1-2	N1-3	N1-4	N1-5	N1-6	N1-7
26-4	0.44	0.32	0.40	0.45	0.60	0.43	0.45	0.45	0.52
25-26	0.58	0.39	0.60	0.65	0.96	0.67	0.72	0.69	1.05
24-4	1.13	0.86	1.40	1.54	2.20	1.44	1.51	1.54	2.28
24-26	0.26	0.18	0.24	0.20	0.28	0.27	0.26	0.28	0.37
236-2	0.66	0.39	0.72	0.73	0.96	0.74	0.79	0.77	1.19
23-4	0.55	0.39	0.60	0.65	0.92	0.67	0.68	0.73	1.01
23-26	0.47	0.32	0.48	0.49	0.64	0.50	0.57	0.57	0.71
25-25	2.74	1.86	3.72	2.76	5.88	3.98	3.82	3.94	6.57
24-25	2.04	1.25	2.52	2.72	4.04	2.71	2.76	2.76	4.56
24-24	0.91	0.61	1.00	1.18	1.64	1.17	1.21	1.18	1.87
23-25	3.03	1.97	2.76	4.26	6.16	4.28	4.23	4.31	8.10
236-3	0.95	0.50	0.36	0.89	1.52	0.37	0.53	0.45	0.67
23-24	1.24	0.79	1.48	1.30	2.04	1.47	1.40	1.50	2.73
26-34	1.09	0.61	1.28	1.30	1.92	1.30	1.36	1.42	2.46
234-2	0.44	0.29	0.52	0.49	0.80	0.53	0.57	0.61	1.01
236-4	1.61	1.00	2.12	2.11	2.92	1.60	2.23	2.24	4.22
23-23	1.06	0.61	1.24	1.26	1.56	1.24	1.36	1.38	2.13
235-4	0.33	0.21	0.36	0.37	0.44	0.30	0.42	0.41	0.63
236-25	1.28	0.82	1.64	1.58	2.40	1.74	1.89	1.87	3.29
245-4	1.86	0.90	2.40	2.32	3.52	2.51	2.46	2.72	4.70
25-34	1.28	0.72	1.36	1.58	2.76	1.47	1.59	1.67	2.13
24-34	3.50	2.01	4.64	4.43	7.24	4.91	4.80	5.16	9.78
235-25	0.80	0.68	0.92	0.85	0.92	0.80	0.98	0.98	1.23
23-34	1.97	1.22	2.68	2.48	3.96	2.81	2.80	3.01	5.75
235-24 or 245-25	1.61	1.00	1.96	1.91	3.04	2.24	2.15	2.36	4.26
234-4	1.39	0.86	1.20	1.62	2.68	1.40	1.81	1.87	3.51
245-24	1.20	0.75	1.40	1.34	2.12	1.50	1.55	1.71	2.99
235-23	0.88	0.54	0.76	1.06	1.60	1.27	1.25	1.54	2.43
234-25	1.24	0.68	0.80	1.30	2.08	1.64	1.59	1.75	3.17
234-24	0.99	0.72	0.96	0.89	1.68	1.34	1.36	1.46	2.35
236-34	2.19	1.11	2.92	2.52	4.44	3.01	3.06	3.33	6.42
234-23	0.95	0.54	0.76	0.77	1.16	1.17	1.21	1.34	2.17
34-34	0.73	0.50	0.76	0.73	1.20	0.87	0.91	1.02	1.57
245-34	1.82	1.11	2.24	1.75	3.20	2.47	2.27	2.85	5.00
234-34	1.82	1.22	1.72	1.99	3.00	2.00	2.31	2.64	4.22
Total PCB	45.04	27.95	50.87	52.47	82.47	56.80	58.84	62.51	107.05

\*\*\* indicates concentration of extract was below lowest calibration standard (0.1 ppm)

\* indicates not found.

Table I. Quantitative Report and QC Data for Polychlorinated Biphenyls in Core Samples  
from Cape Canaveral (Second Sample Set) Service Request SF-0-15.

Concentration µg PCB/g dry soil

File Name:	463N21.TXT	464N22.TXT	465N23.TXT	466N24.TXT	467N25.TXT	468N26.TXT	469N27.TXT
PCB Congener	N2-1	N2-2	N2-3	N2-4	N2-5	N2-6	N2-7
26-4	0.44	0.60	0.36	0.56	0.71	0.68	0.42
25-26	0.64	0.89	0.44	0.80	1.61	1.17	0.59
24-4	1.39	2.01	1.29	1.56	2.89	1.54	1.31
24-26	0.28	0.36	0.24	0.36	0.54	0.45	0.30
236-2	0.76	1.09	0.73	0.88	1.70	1.32	0.72
23-4	0.64	0.68	0.61	0.68	1.31	0.98	0.63
23-26	0.52	0.64	0.44	0.60	0.92	0.75	0.55
25-25	3.58	5.35	3.60	4.23	9.53	6.62	3.33
24-25	2.43	3.70	2.43	2.91	6.67	5.12	2.32
24-24	1.11	1.61	1.09	1.32	2.77	2.14	1.01
23-25	3.98	5.84	3.76	4.55	11.23	7.90	3.67
236-3	0.48	1.37	0.69	0.48	0.69	0.71	0.42
23-24	1.15	1.89	1.01	1.60	3.57	2.63	1.27
26-34	1.27	1.85	1.21	1.44	3.22	2.52	1.22
234-2	0.52	0.80	0.57	0.60	1.25	1.05	0.59
236-4	2.07	3.06	2.02	2.31	6.05	4.33	1.94
23-23	1.27	1.69	1.21	1.40	2.62	2.14	1.22
235-4	0.36	0.52	0.36	0.40	0.80	0.64	0.38
236-25	1.67	2.50	1.62	1.84	4.50	3.01	1.65
245-4	2.39	3.42	2.18	2.43	6.40	4.51	2.28
25-34	1.39	1.89	0.93	1.52	3.46	1.77	1.35
24-34	4.58	6.52	4.37	4.87	13.41	8.69	4.18
235-25	0.99	1.05	0.89	1.00	1.43	1.09	0.97
23-34	2.67	3.70	2.59	2.67	7.57	5.00	2.53
235-24 or 245-25	2.03	2.78	2.02	2.11	5.75	3.65	1.94
234-4	1.71	2.33	1.70	1.88	4.47	3.27	1.65
245-24	1.51	1.97	1.01	1.56	3.90	2.67	1.48
235-23	1.23	1.61	0.85	1.24	3.28	2.14	1.22
234-25	1.51	2.05	1.54	1.60	4.08	2.75	1.56
234-24	1.31	1.73	0.89	1.36	3.07	2.07	1.18
236-34	3.06	3.86	2.99	3.11	8.28	5.23	2.87
234-23	1.19	1.49	0.93	1.24	2.80	1.92	1.22
34-34	0.92	1.05	0.93	0.92	1.91	1.24	0.93
245-34	2.51	3.22	2.43	2.55	6.85	4.18	2.45
234-34	2.35	2.78	2.34	2.43	5.24	3.42	2.32
Total PCB	55.91	77.9	52.3	61.0	144.5	99.3	53.7

\*\*\* Indicates concentration of extract was below lowest calibration standard (0.1 ppm)

\* Indicates not found.



Ref: KPJ 4  
Contract#68-C-98-138  
10-05-98

Dr. Kampbell  
National Risk Management Research Laboratory  
Subsurface Protection and Remediation Division  
U.S. Environmental Protection Agency  
P.O. Box 1198  
Ada, OK 74820

Thru: Dr. Fine *to fine*

Dear Don:

As per service request SF-0-15, the soil samples from Cape Canaveral have been extracted for GCMS analysis and percent moisture has been calculated. For percent moisture calculation, ten grams of soil were and the following equation was used:  
$$(\text{wet soil-dry soil})/(\text{wet soil}) \times 100$$

Sample #	% Moisture
A1-1	9.39
A1-2	9.61
A1-3	10.64
A1-4	10.02
A1-5	10.99
A1-6	9.05
A1-7	10.02
A2-1	10.60
A2-2	10.08
A2-3	11.11
A2-4	10.43
A2-5	10.92
A2-6	12.24
A2-7	10.90
X1-1	12.62
X1-2	13.15
X1-3	13.59
X1-4	12.56
X1-5	16.18

ManTech Environmental Research Services Corporation

R.S. Kerr Environmental Research Laboratory, P.O. Box 1198, 919 Research Drive  
Ada, Oklahoma 74821-1189 405-436-8660 FAX 405-436-8601

Sample#	% Moisture
X1-6	17.72
X1-7	17.26
X2-1	15.29
X2-2	17.83
X2-3	19.53
X2-4	16.11
X2-5	14.97
X2-6	14.30
X2-7	21.06
N1-1	7.13
N1-2	6.26
N1-3	5.59
N1-4	6.17
N1-5	6.85
N1-6	5.94
N1-7	6.37
N2-1	4.08
N2-2	4.47
N2-3	4.03
N2-4	3.96
N2-5	4.04
N2-6	4.59
N2-7	3.75

If you have any questions concerning the information contained in this report, please contact me at your convenience.

Sincerely:



Ken Jewell

xc: R. Cosby  
J. Seeley *JS.*  
D. Fine  
B. Lyon



Ref: 98-PR30/pr  
Contract # 68-C-98-138  
October 5, 1998

Dr. Don Kampbell  
National Risk Management Research Laboratory  
Subsurface Protection and Remediation Division  
U.S. Environmental Protection Agency  
P.O. Box 1198  
Ada, Ok 74820

THRU: D. Fine *dfine*

Dear Don:

Attached is the report for the HPLC analysis of acetic acid and butyric acid for the Cape Canaveral samples as per service request SF-0-15. The samples were analyzed using a Waters 431 Conductivity detector. The samples were analyzed in units of  $\mu\text{M}$ . Check standard (CS  $15\mu\text{M}$ ) and standard (STD  $25\mu\text{M}$ ) were used in the analysis. The samples were analyzed using a sample injection volume of 100  $\mu\text{l}$ . The calibration range was  $5\mu\text{M}$  to  $100\mu\text{M}$ .

A Dionex ICE-AS1 IonPac column and AMMS-ICE MicroMembrane Suppressor were used in the analysis. The suppressor reagent used was 5 mM tetrabutylammonium hydroxide and the eluent was 1.0 mM heptafluorobutyric acid. The flow rate was 0.8 ml/min for the eluent and 5.0 ml/min for the suppressor reagent.

The extraction procedure used is as follows: weigh 3 grams of soil into a flask, add 100 mL of 0.1 M HCl to each flask. Place the samples on a shaker with a rotation rate of 150 cycles per minute for 24 hours. Filter the extract through 0.2  $\mu\text{M}$  syringe filters.

The concentrations of acetic acid and butyric acid are reported as mg/kg and were calculated by the following equation: (mg acid/liter extract) (liter extract/kg dry soil). The dry soil weights were determined by the equation: wet soil weight - (% moisture x wet soil weight). This analysis was performed by Priscilla Rodebush.

Sincerely,

Priscilla Rodebush

XC: R.L. Cosby  
J.L. Seeley  
G.B. Smith *JS*

ManTech Environmental Research Services Corporation

R.S. Kerr Environmental Research Center, P.O. Box 1198, 919 Kerr Research Drive  
Ada, Oklahoma 74821-1198 580-436-8660 FAX 580-436-8501

## MANTECH ENVIRONMENTAL RESEARCH SERVICES CORPORATION

## RESULTS-QC SAMPLES

#	Sample Name	acetic acid (uM)	butyric acid (uM)
1	HPLC BLANK	ND	ND
2	CS 15 uM	13.5	14.7
3	STD 25 uM	25.1	25.7
4	HPLC BLANK	ND	ND
5	STD 25 uM	27.1	26.4

## RESULTS - SAMPLES

#	Sample Name	acetic acid (mg/kg)	butyric acid (mg/kg)
1	A1 4	63.4	ND
2	A2 4	36.2	ND
3	A1 7	16.3	ND
4	A2 7	ND	ND
5	A2 7 dup	ND	ND
6	N1 4	33.9	ND
7	N2 4	83.8	ND
8	X1 4	54.5	ND
9	X2 4	56.0	ND

Dear Don:

Attached are inorganic results for a set of soil samples from Cape Canaveral, FL, submitted to MERSC under Service Request # SF-0-15. The samples were received August 28 and stored at 4° C until October 16 where they were subsampled. The soils were air dried for 48 hours prior to extraction. Soils for pH, chloride and sulfate were extracted into RO water (1:1) and the supernatants analyzed using EPA method 150.1 for pH, and Waters capillary electrophoresis method N-601 for chloride and sulfate. The soils for nitrate were extracted into a 2 molar potassium chloride solution prior to analysis by Lachat FIA method 10-107-04-2-A.

Quality assurance measures performed on this set of samples included spikes, duplicates, known AQC samples and blanks.

If you have any questions concerning this data, please feel free to contact me.

Sincerely,

*Lynda Pennington*  
Lynda Pennington

cc: R.L. Cosby  
J.L. Seeley  
G.B. Smith

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ManTech Environmental Research Services Corporation

R.S. Kerr Environmental Research Center, P.O. Box 1198, 919 Kerr Research Drive  
Ada, Oklahoma 74821-1198 580-436-8660 FAX 580-436-8401

October 22, 1998

SF-0-15  
Don Campbell's  
Cape Canaveral soils

Page 1

SAMPLE	pH	mg Cl <sup>-</sup> (Kg soil)	mg SO <sub>4</sub> <sup>2-</sup> (Kg soil)		SAMPLE	mg NO <sub>3</sub> <sup>-</sup> (Kg soil)
N1-2	8.16	9.84	18.3		N1-1	4.68
N1-5	8.27	9.67	16.8		N1-6	7.04
N2-2	8.10	11.8	18.1		N2-1	4.75
N2-5	(8.17) (8.18)	(9.86) (10.0)	(17.1) (17.3)		N2-6	6.17
X1-2	8.03	1,820	1,480		X1-1	163
X1-5	8.06	1,800	1,270		X1-6	89.6
X2-2	8.06	2,300	1,820		X2-1	154
X2-5	7.99	2,310	1,730		X2-6	175
A1-2	9.03	20.4	80.6		A1-1	5.04
A1-5	9.24	20.5	71.2		A1-6	3.32
A2-2	8.97	12.4	34.2		A2-1	2.12
A2-5	9.01	15.6	45.3		A2-6	2.26
Blank	~~~~~	<1	<1		Blank	<1
WPO39	~~~~~	11.4	62.1		WPO39	1.06
WPO39 T.V.	~~~~~	10.8	58.0		WPO39 T.V.	1.10
Spike Rec.	~~~~~	100%	98%		Spike Rec.	100%

Results from Third Sampling Event  
(Day 117)

Sampling Date: 9-November-1998

R.S. Kerr Laboratory Service Request Number: SF-0-26

**concentrations are mg PCB/g dry soil**

\* congener was not found in extract

**samples rec'd: 16NOV98**

concentrations are mg PCB/g dry soil

File Name:	484A25.TXT	486X11.TXT	487X12.TXT	488X13.TXT	489X14.TXT	490X15.TXT	492X21.TXT	493X22.TXT	494X23.TXT
Sample ID:	A2-5	X1-1	X1-2	X1-3	X1-4	X1-5	X2-1	X2-2	X2-3
PCB Congener	Library #								
26-4	1	1.68	0.47	0.36	0.36	0.44	0.45	0.41	0.46
25-28	2	3.24	0.65	0.40	0.38	0.51	0.65	0.51	0.80
24-4	3	1.17	1.20	0.91	0.90	1.09	1.14	1.02	1.17
24-28	4	1.09	0.25	0.18	0.22	0.22	0.28	0.20	0.25
236-2	5	3.63	0.69	0.51	0.47	0.65	0.72	0.61	0.64
23-4	6	1.84	0.54	0.40	0.40	0.51	0.55	0.47	0.53
23-26	7	1.60	0.51	0.40	0.36	0.44	0.48	0.44	0.46
25-25	8	17.34	3.30	1.89	1.77	2.65	3.58	2.57	3.01
24-25	9	13.63	2.25	1.31	1.26	1.89	2.75	1.83	2.09
24-24	10	5.16	1.02	0.69	0.65	0.91	1.20	0.88	1.03
23-25	11	19.34	3.52	2.11	1.88	2.80	4.41	2.94	3.26
238-3	12	0.82	0.33	0.29	0.25	0.29	0.31	0.20	0.28
23-24	13	0.56	1.27	0.07	0.83	1.02	1.65	1.05	1.24
26-34	14	5.12	1.20	0.80	0.79	0.98	1.55	1.05	1.20
234-2	15	1.52	0.51	0.33	0.32	0.44	0.62	0.41	0.50
236-2	16	10.66	1.85	1.02	0.84	1.63	2.44	1.58	1.83
23-23	17	4.34	1.12	0.80	0.79	0.94	1.38	0.95	1.03
235-4	18	1.06	0.33	0.25	0.25	0.29	0.41	0.34	0.32
236-25	19	6.29	1.49	0.95	0.87	1.13	1.96	1.25	1.31
245-4	20	8.16	1.89	1.16	1.16	1.49	2.78	1.73	1.88
25-34	21	1.29	1.16	0.80	0.76	0.94	1.20	0.98	1.06
24-34	22	16.41	3.81	2.25	1.95	2.87	5.30	3.18	3.47
235-25	23	1.68	0.83	0.73	0.76	0.76	0.96	0.78	0.81
23-34	24	9.69	2.18	1.27	1.23	1.78	3.06	1.83	2.02
235-24 or 245-25	25	8.91	1.83	1.09	0.97	1.27	2.51	1.52	1.52
234-4	26	5.74	1.45	0.87	0.87	1.09	1.79	1.25	1.38
245-24	27	4.80	1.20	0.84	0.83	1.02	1.79	1.05	1.17
236-23	28	3.91	0.98	0.58	0.54	0.80	1.45	0.81	0.85
234-25	29	4.65	1.23	0.84	0.79	1.09	1.79	1.08	1.17
234-24	30	3.55	1.09	0.78	0.78	0.91	1.38	0.88	0.99
236-34	31	9.37	2.32	1.49	1.34	1.78	3.51	2.03	2.16
234-23	32	3.18	0.91	0.89	0.89	0.80	1.20	0.91	0.89
34-34	33	1.76	0.89	0.58	0.58	0.62	0.86	0.68	0.71
245-34	34	7.27	1.89	1.31	1.19	1.42	2.79	1.73	1.77
234-34	35	5.62	1.81	1.38	1.37	1.60	2.48	1.66	1.73
Total PCB		199.88	47.57	31.12	29.46	38.93	61.41	40.77	44.58
									56.20

\*\*\* conc. of extract was less than lowest calibration standard (0.1 ppm)

\* congener was not found in extract

samples rec'd: 16NOV98  
analyses completed: 12DEC98

Analyzed by RWC

concentrations are mg PCB/g dry soil

File Name: Sample ID:		495X24.TXT X2-4	496X25.TXT X2-5	498N11.TXT N1-1	499N12.TXT N1-2	500N13.TXT N1-3	501N14.TXT N1-4	502N18.TXT N1-5	504N21.TXT N2-1	510N21D.TXT N2-1 dup
PCB Congener	Library #									
28-4	1	0.39	0.42	0.40	0.34	0.36	0.33	0.33	0.29	0.29
28-26	2	0.50	0.53	0.79	0.67	0.62	0.59	0.72	0.68	0.69
24-4	3	0.96	1.12	1.80	1.64	1.87	1.52	1.66	1.36	1.32
24-26	4	0.21	0.26	0.32	0.22	0.29	0.22	0.29	0.22	0.26
238-2	5	0.57	0.60	0.97	0.89	1.08	0.78	0.87	0.77	0.73
23-4	6	0.46	0.53	0.79	0.71	0.82	0.67	0.76	0.62	0.62
23-26	7	0.43	0.46	0.58	0.52	0.61	0.56	0.54	0.51	0.51
25-25	8	2.25	2.74	6.25	4.21	5.62	3.85	4.52	3.74	3.52
24-25	9	1.50	1.89	3.74	3.02	3.80	2.66	3.61	2.64	2.64
24-24	10	0.76	0.88	1.55	1.30	1.61	1.15	1.41	1.17	1.14
23-25	11	2.46	3.05	5.87	4.84	6.24	4.33	5.20	4.07	4.10
236-3	12	0.29	0.28	0.43	0.45	0.32	0.37	0.33	0.37	0.37
23-24	13	0.89	1.12	1.91	1.60	2.26	1.46	1.88	1.43	1.47
26-34	14	0.86	1.05	1.40	1.12	1.47	1.18	1.16	0.96	0.96
234-2	15	0.39	0.46	0.81	0.56	0.61	0.56	0.58	0.48	0.51
238-2	16	1.28	1.61	3.06	2.42	3.26	2.26	2.82	2.16	2.16
23-23	17	0.86	1.02	1.40	1.34	1.58	1.26	1.45	1.25	3.81
236-4	18	0.29	0.32	0.43	0.41	0.47	0.33	0.40	0.37	0.37
236-25	19	1.11	1.37	2.12	1.75	2.37	1.67	2.06	1.72	1.65
245-4	20	1.29	1.58	2.68	2.57	3.30	2.37	2.82	2.31	2.31
25-34	21	0.89	1.16	1.37	1.34	1.72	1.67	1.45	1.25	1.28
24-34	22	2.64	3.23	6.97	4.85	6.71	4.66	5.64	4.36	4.21
235-25	23	0.70	0.81	0.97	0.97	1.00	0.93	0.94	0.80	0.88
23-34	24	1.54	1.93	3.20	2.67	3.77	2.85	3.22	2.53	2.45
235-24 or 245-25	25	1.18	1.47	2.59	2.23	2.80	2.07	2.42	1.94	1.98
234-4	26	1.04	1.23	2.12	1.90	2.44	1.74	1.99	1.65	1.65
245-24	27	0.96	1.09	1.67	1.60	2.01	1.41	1.70	1.39	1.43
235-23	28	0.88	0.84	1.48	1.27	1.61	1.15	1.30	1.28	1.17
234-25	29	0.96	1.05	1.84	1.49	2.15	1.44	1.70	1.50	1.47
234-24	30	0.82	0.95	1.37	1.27	1.54	1.15	1.30	1.25	1.21
236-34	31	1.71	1.93	3.38	3.09	4.12	2.92	3.40	2.82	2.67
234-23	32	0.79	0.88	1.37	1.15	1.40	1.04	1.19	1.14	1.10
34-34	33	0.61	0.67	0.94	0.86	1.04	0.81	0.90	0.84	0.88
245-34	34	1.39	1.54	2.81	2.38	3.23	2.37	2.76	2.16	2.23
234-34	35	1.54	1.58	2.52	2.20	2.87	2.18	2.35	2.12	2.12
Total PCB		35.18	41.59	70.19	60.13	77.08	56.50	65.62	54.10	56.05

\*\*\* conc. of extract was less than lowest calibration standard (0.1 ppm)  
\* congener was not found in extract

samples rec'd: 16NOV98  
analyses completed: 12DEC98

concentrations are mg PCB/g dry soil

File Name: Sample ID:	505N22.TXT N2-2	506N23.TXT N2-3	507N24.TXT N2-4	508N25.TXT N2-5					
PCB Congener	Library #								
26-4	1	0.36	0.37	0.33	0.37				
25-26	2	0.75	0.67	0.66	0.66				
24-4	3	1.69	1.42	1.49	1.50				
24-26	4	0.29	0.28	0.26	0.29				
236-2	5	0.97	0.79	0.87	0.80				
23-4	6	0.79	0.60	0.69	0.70				
23-26	7	0.61	0.52	0.58	0.61				
25-25	8	4.77	3.76	4.26	3.91				
24-25	9	3.34	2.70	3.02	2.67				
24-24	10	1.51	1.20	1.35	1.24				
23-25	11	5.38	4.09	4.77	4.39				
236-3	12	0.39	0.41	0.33	0.40				
23-24	13	1.83	1.36	1.68	1.46				
26-34	14	1.33	1.12	1.09	1.13				
234-2	15	0.61	1.42	0.55	0.55				
236-2	16	2.91	2.02	2.69	2.23				
23-23	17	1.51	3.60	4.52	1.24				
235-4	18	0.43	0.41	0.40	0.40				
236-25	19	2.15	1.72	1.93	1.79				
245-4	20	2.98	2.29	2.77	2.34				
25-34	21	1.65	1.35	1.46	1.17				
24-34	22	5.95	4.35	5.32	4.78				
235-25	23	0.90	0.94	0.91	0.88				
23-34	24	3.37	2.59	3.17	2.82				
235-24 or 245-25	25	2.69	2.02	2.40	2.09				
234-4	26	2.22	1.72	2.08	1.78				
245-24	27	1.83	1.50	1.75	1.57				
235-23	28	1.72	1.35	1.53	1.35				
234-25	29	2.04	1.57	1.75	1.65				
234-24	30	1.54	1.18	1.46	1.39				
236-34	31	3.84	2.96	3.42	3.04				
234-23	32	1.40	1.18	1.35	1.21				
34-34	33	1.00	0.90	0.96	0.91				
245-34	34	3.18	2.47	2.84	2.67				
234-34	35	2.76	2.29	2.48	2.41				
Total PCB		70.63	59.08	67.01	58.28				

\*\*\* conc. of extract was less than lowest calibration standard (0.1 ppm)  
 \* congener was not found in extract

samples rec'd: 16NOV98  
 analyses completed: 12DEC98



Ref: KPJ 5  
Contract #68-C3-138  
November 30, 1998

Dr. Kampbell  
National Risk Management Research Laboratory  
Subsurface Protection and Remediation Division  
U.S. Environmental Protection Agency  
P.O. Box 1198  
ADA, OK 74820

Thru: Dr. Fine *Fine*

Dear Don:

As per service request SF-0-26, the soil samples from Cape Canaveral have been extracted for GCMS analysis and percent moisture has been calculated. For percent moisture calculation, ten grams of soil were used and the following equation was applied:  $(\text{wet soil} - \text{dry soil}) / (\text{wet soil}) \times 100$

Samples #	% Moisture
A1-1	3.03
A1-2	3.01
A1-3	3.19
A1-4	3.13
A1-5	3.06
A2-1	2.85
A2-2	3.15
A2-3	3.33
A2-4	2.82
A2-5	2.54
X1-1	7.33
X1-2	6.59
X1-3	7.25
X1-4	7.15
X1-5	7.81
X2-1	6.84
X2-2	7.20
X2-3	7.65
X2-4	7.78
X2-5	6.89
N1-1	2.65
N1-2	1.92
N1-3	1.80
N1-4	1.72

ManTech Environmental Research Services Corporation

R.S. Kerr Environmental Research Center, P.O. Box 1198, 919 Kerr Research Drive  
Ada, Oklahoma 74821-1198 580-436-8660 FAX 580-436-8501


Sample#	% moisture
N1-5	1.50
N2-1	0.80
N2-2	0.56
N2-3	0.66
N2-4	0.43
N2-5	0.43

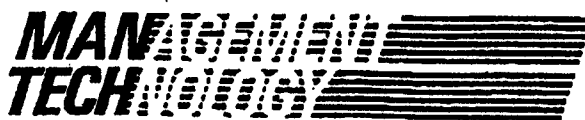
If you have any questions concerning the information contained in this report, please contact me at your convenience.

Sincerely:



Ken Jewell

xc: R. Cosby  
J. Seeley   
D. Fine



Ref: 98-PR54/pr  
Contract # 68-C-98-138  
December 3, 1998

Dr. Don Kampbell  
National Risk Management Research Laboratory  
Subsurface Protection and Remediation Division  
U.S. Environmental Protection Agency  
P.O. Box 1198  
Ada, Ok 74820

THRU: D. Fine *DF*

Dear Don:

Attached is the report for the HPLC analysis of acetic acid and butyric acid for the Cape Canaveral samples as per service request SF-0-26. The samples were analyzed using a Waters 431 Conductivity detector. The samples were analyzed in units of  $\mu\text{M}$ . Check standard (CS 120 $\mu\text{M}$ ) and standards (STD 25 $\mu\text{M}$ ) and (STD 200 $\mu\text{M}$ ) were used in the analysis. The samples were analyzed using a sample injection volume of 100  $\mu\text{l}$ . The calibration range was 5 $\mu\text{M}$  to 100 $\mu\text{M}$ .

A Dionex ICE-AS1 IonPac column and AMMS-ICE MicroMembrane Suppressor were used in the analysis. The suppressor reagent used was 5 mM tetrabutylammonium hydroxide and the eluent was 1.0 mM heptafluorobutyric acid. The flow rate was 0.8 ml/min for the eluent and 5.0 ml/min for the suppressor reagent.

The extraction procedure used is as follows: weigh 3 grams of soil into a flask, add 100 mL of 0.1 M HCl to each flask. Place the samples on a shaker with a rotation rate of 150 cycles per minute for 24 hours. Filter the extract through 0.2  $\mu\text{M}$  syringe filters.

Sincerely,

Priscilla Rodebush

xc: R.L. Cosby

J.L. Seeley *JLS*

G.B. Smith

ManTech Environmental Research Services Corporation

R.S. Kerr Environmental Research Center, P.O. Box 1198, 919 Kerr Research Drive

## MANTECH ENVIRONMENTAL RESEARCH SERVICES CORPORATION

SR# SF-0-26

Originator: Dr. Don Kampbell

Sample Site: Cape Canaveral Air Station

Date Analyzed: December 1 and 2, 1998

Sample Name	Acetic Acid (uM)	Butyric Acid (uM)
HPLC BLANK	ND	ND
CS 120 uM	128	113
STD 25 uM	27.5	23.4
A1 4	ND	ND
A2 4	ND	ND
X1 4	ND	ND
X2 4	ND	ND
N1 4	ND	ND
N2 4	ND	ND
BLANK	ND	ND
HPLC BLANK	ND	ND
STD 200 uM	219	209

ND = non detected

December 17, 1998  
 Rec'd 12-10-98  
 Analyzed 12-16-98 by  
 RJK: NO3  
 LKP: pH, Cl, SO4

SF-0-26  
 Don Kampbell's  
 Cape Canaveral soils

Page 1

SAMPLE	pH	mg Cl <sup>-</sup> (Kg soil)	mg SO <sub>4</sub> <sup>2-</sup> (Kg soil)		SAMPLE	mg NO <sub>3</sub> <sup>-</sup> (Kg soil)
N1-2	8.07	16.5	28.4		N1-1	<1.00
N1-5	8.09	50.0	36.5		N1-4	<1.00
N2-2	8.14	41.6	25.6		N2-1	<1.00
N2-5	8.12	100	23.3		N2-4	<1.00
X1-2	(8.06) (8.13)	871	805		X1-1	(63.2) (65.7)
X1-5	(8.20) (8.20)	(1,090) (1,080)	(927) (914)		X1-4	53
X2-2	8.19	808	638		X2-1	18.8
X2-5	8.30	95.8	309		X2-4	20.1
A1-2	9.94	48.4	55.0		A1-1	<1.00
A1-5	9.90	61.8	61.2		A1-4	<1.00
A2-2	9.77	64.1	49.2		A2-1	<1.00
A2-4	9.67	46.5	44.2		A2-4	<1.00
Blank	~~~~	<1.0	<1.0		Blank	<1.00
WPO39	~~~~	10.8	60.2		WPO39	1.08
WPO39 T.V.	~~~~	10.8	58.0		WPO39 T.V.	1.10
Spike Rec.	~~~~	100%	99%		Spike Rec.	100%

December 30, 1998  
Rec'd 12-10-98  
Analyzed 12-29-98 by  
LKP: Cl, SO<sub>4</sub>

SF-0-26  
Don Kampbell's  
Cape Canaveral soils

Page 2

Analyzed 12-29-98			
Sample		mg Cl <sup>-</sup>	mg SO <sub>4</sub> <sup>-2</sup>
		(Kg soil)	(Kg soil)
X2-2-A		732	786
X2-2-B		841	852
X2-2-C		749	769
X2-5-A		87.5	314
X2-5-B		85.3	296
X2-5-C		73.5	257
WPO39		10.8	58.3
WPO39 T.V.		10.8	58

Results from Fourth Sampling Event  
(Day 202)

Sampling Date: 2-February-1999

R.S. Kerr Laboratory Service Request Number: SF-0-53

23MAR99

SF-0-53 / Kampbell / Cape Canaveral / Polychlorinated Biphenyls (PCBs)

Date Received: 03FEB99

Date Extracted: 12FEB99

Date(s) Analyzed: 22FEB99 to 04MAR99

Analyzed by RWC

File Name:		563BLKIS.TXT	564A11.TXT	565A12.TXT	566A13.TXT	567A14.TXT	568A15.TXT	569_11.TXT	570A21.TXT	571A22.TXT
Sample I.D.								PCB Chk Std		
PCB Congener	Lib #	Blank	A 1-1	A 1-2	A 1-3	A 1-4	A 1-5	1 ng/ul Mix 1	A 2-1	A 2-2
26-4	1	***	0.40	0.27	0.91	0.31	0.54	***	1.45	1.32
25-26	2	***	1.05	1.18	1.39	1.07	0.63	***	2.67	2.43
24-4	3	***	0.65	0.44	0.87	0.55	0.57	0.86	0.74	0.82
24-26	4	***	0.54	0.53	0.58	0.59	0.44	***	0.91	0.76
236-2	5	***	1.62	1.77	1.49	1.94	0.86	***	3.75	3.45
23-4	6	***	0.94	0.86	0.94	0.93	0.51	0.90	1.49	1.55
23-26	7	***	0.54	0.62	0.58	0.73	0.25	***	1.15	1.18
25-25	8	***	9.16	11.33	10.30	9.06	4.56	0.98	20.41	17.96
24-25	9	***	5.48	6.61	5.05	5.77	2.76	***	13.11	11.97
24-24	10	***	2.67	2.48	2.43	2.49	1.43	***	4.29	4.14
23-25	11	***	8.55	10.15	8.65	8.19	3.93	0.77	17.74	18.12
236-3	12	***	0.47	0.50	0.71	0.62	0.44	***	1.08	0.79
23-24	13	***	2.56	3.24	2.69	3.21	1.52	***	5.54	5.26
26-34	14	***	1.62	1.62	1.91	1.90	1.08	0.84	4.16	3.88
234-2	15	***	0.61	0.68	0.71	0.66	0.44	***	1.35	1.45
236-4	16	***	3.86	5.01	4.05	4.63	2.03	***	8.01	7.93
23-23	17	***	1.88	2.01	1.75	1.76	0.95	***	4.02	3.78
235-4	18	***	0.47	0.53	0.52	0.59	0.38	***	0.84	0.89
236-25	19	***	2.89	3.10	2.75	2.87	1.58	***	5.47	5.49
245-4	20	***	4.11	4.25	4.76	4.25	2.15	0.86	8.38	8.55
25-34	21	***	0.47	0.44	0.55	0.48	0.22	***	0.88	1.02
24-34	22	***	8.15	9.00	8.23	8.47	4.15	0.96	15.58	17.24
235-25	23	***	0.79	0.86	0.81	0.93	0.67	***	1.18	1.12
23-34	24	***	4.65	5.10	3.95	4.77	2.19	0.94	9.29	9.18
235-24 or 245-25	25	***	3.10	3.54	3.43	3.56	1.65	***	6.62	6.84
234-4	26	***	2.71	3.24	2.75	2.83	2.12	***	5.27	5.79
245-24	27	***	1.59	2.09	1.98	1.83	1.05	0.75	3.62	3.91
245-23	28	***	1.73	2.06	1.81	1.80	0.98	***	3.21	3.19
234-25	29	***	2.02	2.30	2.20	2.28	1.01	0.93	4.36	4.14
234-24	30	***	1.37	1.53	1.49	1.28	0.67	***	2.50	2.73
236-34	31	***	3.54	4.22	3.56	3.46	1.87	0.73	6.96	6.35
234-23	32	***	1.15	1.30	1.26	1.21	0.73	0.81	1.99	2.01
34-34	33	***	0.97	0.88	0.94	0.97	0.70	***	1.32	1.38
245-34	34	***	3.14	2.65	2.88	2.87	1.71	***	5.20	5.62
234-34	35	*	2.09	2.12	2.11	2.45	1.30	*	4.29	4.31
Total PCB		*	87.57	98.53	91.01	91.32	48.06	10.90	178.86	176.57

\*\*\* indicates extract conc. &lt; low calib. std. (0.1 ng/ul)

\* indicates compound was not detected in extract

Date Received: 03FEB99

Date Extracted: 12FEB99

Date(s) Analyzed: 22FEB99 to 04MAR99

Analyzed by RWC

File Name:		572A23.TXT	573A24.TXT	574A25.TXT	575_21.TXT	576X11.TXT	577X12.TXT	578X13.TXT	579X14.TXT	580X15.TXT
Sample I.D.					PCB Chk Std					
PCB Congener	Lib #	A 2-3	A 2-4	A 2-5	1 ng/ul Mix 2	X 1-1	X 1-2	X 1-3	X 1-4	X 1-5
26-4	1	0.92	1.46	1.44	0.99	0.16	0.20	0.23	0.21	0.22
25-26	2	1.87	2.58	2.77	***	0.25	0.30	0.33	0.31	0.35
24-4	3	0.57	0.88	0.84	***	0.53	0.63	0.63	0.62	0.70
24-26	4	0.81	0.88	0.97	***	0.35	0.39	0.36	0.38	0.35
236-2	5	2.54	3.02	3.44	0.94	0.47	0.53	0.50	0.51	0.47
23-4	6	1.31	1.46	1.47	***	0.41	0.49	0.43	0.48	0.51
23-26	7	0.81	1.26	1.20	0.92	0.06	0.10	0.13	0.14	0.13
25-25	8	15.62	18.12	21.25	***	1.60	2.01	2.15	2.23	2.47
24-25	9	8.87	12.35	12.70	***	1.29	1.51	1.52	1.65	1.64
24-24	10	3.22	4.14	4.58	***	0.75	0.86	0.86	0.86	0.92
23-25	11	12.83	16.60	18.68	***	1.73	2.47	2.35	2.09	2.34
236-3	12	0.85	0.92	1.30	***	0.35	0.43	0.43	0.45	0.44
23-24	13	4.03	5.09	5.45	***	0.85	1.02	1.06	1.06	1.11
26-34	14	3.07	4.28	5.08	***	0.75	0.89	0.89	0.89	0.89
234-2	15	1.02	1.39	1.44	0.89	0.38	0.43	0.43	0.48	0.44
236-4	16	6.57	8.45	8.12	***	1.13	1.28	1.36	1.34	1.58
23-23	17	3.18	3.63	3.57	***	0.60	0.72	0.76	0.72	0.70
235-4	18	0.78	0.78	0.84	***	0.28	0.33	0.33	0.31	0.35
236-25	19	4.59	6.31	7.05	0.90	0.85	0.99	1.03	1.00	1.14
245-4	20	5.87	7.43	8.59	***	1.13	1.18	1.36	1.30	1.30
25-34	21	0.71	1.09	1.27	0.90	0.53	0.56	0.56	0.55	0.66
24-34	22	13.00	16.26	17.97	***	1.92	2.40	2.58	2.61	2.59
235-25	23	1.10	1.15	1.24	***	0.60	0.59	0.63	0.62	0.60
23-34	24	6.89	9.03	9.82	***	1.29	1.41	1.62	1.61	1.68
235-24 or 245-25	25	5.09	6.86	8.19	***	1.01	1.12	0.99	1.13	1.30
234-4	26	4.66	5.74	5.81	0.91	0.94	0.92	1.03	0.96	1.04
245-24	27	2.93	3.87	3.88	***	0.60	0.72	0.76	0.75	0.73
245-23	28	2.72	3.26	3.81	***	0.72	0.79	0.83	0.72	0.79
234-25	29	3.92	3.97	4.88	***	0.72	0.66	0.89	0.79	0.82
234-24	30	2.33	2.61	2.77	***	0.41	0.49	0.53	0.58	0.57
236-34	31	6.11	7.06	8.45	***	1.16	1.41	1.49	1.44	1.61
234-23	32	1.66	1.93	2.07	***	0.53	0.56	0.63	0.62	0.70
34-34	33	1.20	1.39	1.37	***	0.60	0.66	0.69	0.69	0.73
245-34	34	4.03	4.99	5.38	***	1.26	1.38	1.49	1.37	1.42
234-34	35	3.50	3.90	4.71	*	0.97	1.05	1.19	1.13	1.11
Total PCB		139.19	174.15	192.37	7.03	27.20	31.49	33.06	32.60	34.36

\*\*\* Indicates extract conc. &lt; low calib. std. (0.1 ng/ul)

\* Indicates compound was not detected in extract

Date Received: 03FEB99

Date Extracted: 12FEB99

Date(s) Analyzed: 22FEB99 to 04MAR99

Analyzed by RWC

File Name		581_31.TXT	582A25D.TXT	583X21.TXT	584X22.TXT	585X23.TXT	586X24.TXT	587_41.TXT	588X25.TXT	589N11.TXT
Sample I.D.		PCB Chk Std						PCB Chk Std		
PCB Congener	Lib #	1 ng/ul Mix 3	A 2-5 dup	X 2-1	X 2-2	X 2-3	X 2-4	1 ng/ul Mix 4	X 2-5	N 1-1
26-4	1	***	1.33	0.15	0.24	0.14	0.16	***	0.22	0.11
25-26	2	***	2.22	0.36	0.32	0.22	0.29	0.88	0.28	0.50
24-4	3	***	0.86	0.58	0.59	0.42	0.61	***	0.62	1.21
24-26	4	***	0.89	0.30	0.35	0.30	0.35	0.80	0.34	0.43
236-2	5	***	3.08	0.49	0.47	0.33	0.48	***	0.48	0.92
23-4	6	***	1.37	0.43	0.44	0.36	0.48	***	0.42	0.74
23-26	7	***	1.13	0.12	0.12	0.11	0.13	***	0.14	0.35
25-25	8	***	15.16	1.70	1.74	1.44	1.96	***	1.91	4.68
24-25	9	***	11.09	1.31	1.39	1.00	1.41	***	1.38	3.05
24-24	10	0.90	3.83	0.79	0.80	0.69	0.80	***	0.79	1.42
23-25	11	***	15.06	2.28	2.19	1.63	2.02	***	2.08	4.75
236-3	12	***	0.99	0.36	0.35	0.33	0.42	***	0.37	0.57
23-24	13	0.87	4.79	0.94	0.97	0.86	0.90	***	0.90	1.81
26-34	14	***	4.14	0.76	0.77	0.66	0.77	***	0.76	1.03
234-2	15	***	1.40	0.40	0.41	0.30	0.42	***	0.28	0.60
236-4	16	***	8.08	1.25	1.12	0.94	1.32	***	1.26	2.73
23-23	17	0.90	3.63	0.55	0.62	0.55	0.71	***	0.70	1.24
235-4	18	***	0.82	0.30	0.32	0.25	0.32	***	0.28	0.43
236-25	19	***	5.51	0.94	0.95	0.72	0.90	***	0.84	1.77
245-4	20	***	7.70	1.16	1.03	0.89	1.09	***	1.15	3.12
25-34	21	***	0.99	0.49	0.47	0.39	0.58	***	0.53	0.92
24-34	22	***	15.23	2.07	2.48	1.72	2.02	***	2.30	5.49
235-25	23	0.82	1.13	0.55	0.53	0.50	0.58	***	0.51	0.78
23-34	24	***	8.32	1.49	1.48	1.05	1.28	***	1.29	3.26
235-24 or 245-25	25	0.86	5.24	1.22	1.03	0.86	1.09	0.98	0.98	2.48
234-4	26	***	4.52	0.94	0.89	0.80	0.90	***	0.82	1.91
245-24	27	*	3.35	0.64	0.62	0.50	0.64	***	0.62	1.35
245-23	28	***	3.42	0.82	0.71	0.53	0.64	***	0.73	1.31
234-25	29	***	3.49	0.79	0.74	0.53	0.71	***	0.67	1.70
234-24	30	***	2.46	0.40	0.50	0.30	0.42	***	0.42	0.89
236-34	31	***	7.15	1.16	1.24	0.89	1.19	***	1.12	2.62
234-23	32	***	1.98	0.55	0.50	0.44	0.58	***	0.48	0.96
34-34	33	***	1.51	0.61	0.62	0.53	0.67	***	0.56	0.89
245-34	34	0.87	5.03	1.16	1.27	1.08	1.22	***	1.21	1.95
234-34	35	0.79	3.80	0.97	0.97	0.91	1.00	*	0.98	1.56
Total PCB		6.41	160.70	29.01	29.27	23.18	29.05	2.93	28.44	59.52

\*\*\* indicates extract conc. &lt; low calib. std. (0.1 ng/ul)

\* Indicates compound was not detected in extract

Date Received: 03FEB99

Date Extracted: 12FEB99

Date(s) Analyzed: 22FEB99 to 04MAR99

Analyzed by RWC

File Name		590N12.TXT	591N13.TXT	592N14.TXT	593_51.TXT	594N15.TXT	595N21.TXT	596N22.TXT	597N23.TXT	598N24.TXT
Sample I.D.					PCB Chk Std					
PCB Congener	Lib #	N 1-2	N 1-3	N 1-4	1 ng/ul Mtx 5	N 1-5	N 2-1	N 2-2	N 2-3	N 2-4
26-4	1	0.11	0.07	0.07	***	0.07	0.08	0.04	0.08	0.05
25-26	2	0.51	0.40	0.48	***	0.37	0.38	0.34	0.38	0.15
24-4	3	0.88	0.92	0.92	***	1.03	0.95	0.73	0.95	0.40
24-26	4	0.44	0.40	0.44	***	0.37	0.42	0.42	0.42	0.12
236-2	5	0.80	0.73	0.74	***	0.73	0.73	0.65	0.69	0.26
23-4	6	0.69	0.62	0.66	***	0.62	0.57	0.57	0.69	0.20
23-26	7	0.29	0.22	0.22	***	0.18	0.15	0.15	0.19	0.08
25-25	8	3.98	3.30	3.90	***	3.30	3.17	3.09	3.32	1.35
24-25	9	3.32	2.60	2.95	0.90	2.45	2.67	2.02	2.41	0.98
24-24	10	1.39	1.21	1.29	***	1.10	1.18	1.11	1.18	0.41
23-25	11	4.49	3.89	4.09	***	3.52	3.51	3.28	3.63	1.37
236-3	12	0.47	0.59	0.55	0.97	0.51	0.50	0.46	0.53	0.17
23-24	13	1.68	1.50	1.40	***	1.50	1.41	1.22	1.41	0.49
26-34	14	0.95	0.92	0.88	***	0.88	0.92	0.80	0.95	0.29
234-2	15	0.55	0.55	0.55	***	0.48	0.53	0.46	0.46	0.17
236-4	16	2.19	1.87	2.25	0.81	1.90	1.91	1.64	1.91	0.67
23-23	17	1.09	1.03	1.07	***	0.95	1.03	0.84	1.03	0.30
235-4	18	0.44	0.40	0.44	0.82	0.40	0.42	0.38	0.38	0.12
236-25	19	1.90	1.65	1.66	***	1.39	1.53	1.53	1.49	0.49
245-4	20	2.55	2.13	2.43	***	1.98	2.02	1.83	2.02	0.72
25-34	21	0.88	0.84	0.88	***	0.84	0.92	0.80	1.03	0.39
24-34	22	5.18	4.51	4.49	***	4.28	4.96	3.86	4.62	1.72
235-25	23	0.73	0.70	0.74	***	0.70	0.76	0.76	0.73	0.20
23-34	24	2.85	2.57	2.62	***	2.34	2.18	1.95	2.44	0.73
235-24 or 245-25	25	1.90	1.98	1.99	***	1.90	2.06	1.79	1.87	0.59
234-4	26	1.90	1.54	1.73	***	1.50	1.53	1.45	1.76	0.50
245-24	27	1.20	1.14	1.22	***	0.95	1.18	0.88	1.22	0.35
245-23	28	1.35	1.06	1.25	0.91	1.10	1.11	0.99	1.03	0.36
234-25	29	1.39	1.03	1.36	***	1.24	1.15	1.22	1.07	0.38
234-24	30	0.95	0.73	0.81	1.11	0.70	0.76	0.69	0.69	0.22
236-34	31	2.30	1.83	2.25	***	1.94	1.76	1.76	2.22	0.66
234-23	32	0.95	0.84	0.81	***	0.77	0.80	0.73	0.80	0.22
34-34	33	0.88	0.84	0.85	0.95	0.81	0.80	0.80	0.88	0.22
245-34	34	2.26	1.83	2.03	***	1.83	1.68	1.76	2.06	0.57
234-34	35	1.46	1.47	1.51	*	1.57	1.49	1.49	1.57	0.43
Total PCB		54.86	47.93	51.53	6.90	46.21	47.24	42.50	48.13	16.33

\*\*\* indicates extract conc. &lt; low calib. std. (0.1 ng/ul)

\* indicates compound was not detected in extract

23MAR99

SF-0-53 / Kampbell / Cape Canaveral / Polychlorinated Biphenyls (PCBs)

Date Received: 03FEB99

Date Extracted: 12FEB99

Date(s) Analyzed: 22FEB99 to 04MAR99

Analyzed by RWC

File Name		599_3P5.TXT	600N25.TXT	601X25D.TXT
Sample I.D.		PCB Chk Std		
PCB Congener	Lib #	0.5 ng/ul Mix 3	N 2-5	X 2-5 dup
26-4	1	***	0.08	0.15
25-26	2	***	0.42	0.24
24-4	3	***	1.07	0.54
24-26	4	***	0.46	0.33
236-2	5	***	0.76	0.39
23-4	6	***	0.65	0.39
23-26	7	***	0.27	0.15
25-25	8	***	3.84	1.70
24-25	9	***	2.81	1.25
24-24	10	0.43	1.26	0.72
23-25	11	***	3.99	1.82
236-3	12	***	0.61	0.36
23-24	13	0.45	1.41	0.89
26-34	14	***	1.03	0.74
234-2	15	***	0.53	0.39
236-4	16	***	2.09	1.04
23-23	17	0.44	1.10	0.63
235-4	18	***	0.42	0.27
236-25	19	***	1.79	0.80
245-4	20	***	2.62	0.98
25-34	21	***	1.10	0.48
24-34	22	***	4.87	1.82
235-25	23	0.41	0.76	0.51
23-34	24	***	2.78	1.10
235-24 or 245-25	25	0.44	1.98	0.98
234-4	26	***	1.79	0.77
245-24	27	***	1.26	0.66
245-23	28	***	1.26	0.63
234-25	29	***	0.99	0.63
234-24	30	***	0.68	0.36
236-34	31	***	2.24	1.16
234-23	32	***	0.76	0.48
34-34	33	***	0.91	0.60
245-34	34	0.51	2.13	1.19
234-34	35	0.51	1.67	0.92
Total PCB		3.71	52.38	26.04

\*\*\* indicates extract conc. &lt; low calib. std (0.1 ng/ul)

\* indicates compound was not detected in extract



# MEMORANDUM

**MANTECH ENVIRONMENTAL RESEARCH SERVICES CORP.**  
Environmental Science

**In reply refer to :99-KPJ2  
Contract # 68-C-98-138**

To: Dr. Kampbell

**From:** Ken Jewell

Thru: D.D. Fine

Date: 2-18-99

**Subject: Report letter for SF-0-53.**

**Copies:** R.L. Cosby,

Ref: 99-KPJ-2

**J.L. Seeley**

G.B. Smith

As per service request SF-0-53, the soil samples from Cape Canaveral have been extracted for GCMS analysis and percent moisture has been calculated. For percent moisture calculation, ten grams of soil were used and the following equation was applied:

$$\frac{(\text{Wet soil} - \text{Dry soil})}{(\text{Wet soil})} \times 100$$

Sample#	%Moisture
A1-1	14.39
A1-2	25.63
A1-3	23.79
A1-4	16.25
A1-5	14.64
A2-1	14.76
A2-2	15.81
A2-3	15.72
A2-4	16.00
A2-5	14.99
X1-1	19.71
X1-2	17.34
X1-3	18.45
X1-4	17.08
X1-5	16.95
X2-1	22.66
X2-2	22.65
X2-3	24.33

**ManTech Environmental Research Services Corporation**

**R.S. Kerr Environmental Research Laboratory, P.O. Box 1198, 919 Research Drive  
Ada, Oklahoma 74821-1198 405-436-8660 FAX 405-436-8501**

X2-4

22.50

Sample #	% Moisture
X2-5	24.80
N1-1	8.28
N1-2	8.55
N1-3	8.27
N1-4	7.52
N1-5	Sample container arrived broken.
N2-1	3.27
N2-2	4.49
N2-3	3.27
N2-4	2.49
N2-5	2.41

If you have any questions concerning the information contained in this report, please contact me at your convenience.



Ken Jewell

xc: R. Cosby  
J. Seeley  
D. Fine

MEMORANDUM

MANTECH ENVIRONMENTAL RESEARCH SERVICES CORP.

Environmental Science

In reply refer to: 99-PR18  
Contract # 68-C-98-138

To: Dr. Don Kampbell

From: Priscilla Rodebush PR

Thru: Dr. Dennis Fine *dfine*

Subject: SF-0-53

Date: March 12, 1999

Copies: J.L. Seeley *JS*  
R.L. Cosby  
G.B. Smith

Attached is the report for the HPLC analysis of acetic acid and butyric acid for the Cape Canaveral samples as per service request SF-0-53. The samples were analyzed using a Waters 431 Conductivity detector. The samples were analyzed in units of  $\mu\text{M}$ . Check standard (CS 120 $\mu\text{M}$ ) and standard (STD 200 $\mu\text{M}$ ) were used in the analysis. The samples were analyzed using a sample injection volume of 50  $\mu\text{l}$ . The calibration range was 10 $\mu\text{M}$  to 600 $\mu\text{M}$ .

A Dionex ICE-ASI IonPac column and AMMS-ICE MicroMembrane Suppressor were used in the analysis. The suppressor reagent used was 5 mM tetrabutylammonium hydroxide and the eluent was 1.0 mM heptafluorobutyric acid in 10% acetonitrile and 90% deionized water. The flow rate was 0.8 ml/min for the eluent and 5.0 ml/min for the suppressor reagent.

The extraction procedure used is as follows: weigh 3 grams of soil into a flask, add 100 mL of 0.1 M HCl to each flask. Place the samples on a shaker with a rotation rate of 150 cycles per minute for 24 hours. Filter the extract through 0.2  $\mu\text{M}$  syringe filters.

The concentrations of acetic and butyric acids were all non-detected, therefore no calculations were made. The analysis was performed by Priscilla Rodebush.

Service Request: SF-0-53  
Originator: Dr. Don Kampbell  
Sample Site: Cape Canaveral  
Date Received: 2/3/99  
Date Analyzed: 3/10/99  
Analyst: Priscilla Rodebush

SampleName	Acetic Acid (uM)	Butyric Acid (uM)
CS 120 uM	123	128
STD 200 uM	192	219
HPLC BLANK	nd	nd
N2-3	nd	nd
N2-3	nd	nd
N1-3	nd	nd
A1-3	nd	nd
A2-3	nd	nd
A1-5	nd	nd
A2-5	nd	nd
X1-3	nd	nd
X2-3	nd	nd
HPLC BLANK	nd	nd
CS 120 uM	111	127
STD 200 uM	205	220

nd = non detected

**MEMORANDUM****MANTECH ENVIRONMENTAL RESEARCH SERVICES CORP.**  
**Environmental Science**

In reply refer to: 99-17LP/lp  
Contract # 68-C-98-138

To: ~~Dr. Don Campbell~~

Thru: D.D. Fine *DDF*

From: Lynda Pennington *LP*

Subject: SR # SF-0-53

Ref:

Copies: R.L. Cosby

G.B. Smith

J.L. Seeley *WEL for JLS*

Date: March 9, 1999

Attached are inorganic results for 24 Cape Canaveral, Florida soil samples submitted to MERSC under Service Request # SF-0-53. The samples were received February 3, stored in the refrigerator until a portion was removed from each sample and air dried on March 1, and were analyzed March 8 and 9, 1999. The soils were extracted 1:1 with RO water for pH, chloride and sulfate and 1:2 with 2 M potassium chloride for nitrate analysis. The methods used were Waters capillary electrophoresis method N-601 for chloride and sulfate; Lachat FIA methods 10-107-04-2-A for nitrate; and EPA method 150.1 for pH.

Quality control measures performed along with your samples included analysis of blanks, duplicates, spikes, known WPO samples and check standards.

If you have any questions concerning this data, please feel free to contact me.

Rec'd Feb. 3, 1999

Analyzed March 8 and 9

SF-0-53

Don Kampbell  
Cape Canaveral, FL

Page 1

SAMPLE	pH	mg Cl <sup>-</sup> per Kg soil	mg SO <sub>4</sub> <sup>2-</sup> per Kg soil
N1-2	8.13	5.21	14.6
N1-5	8.12	18.2	43.9
X1-2	8.09	1,230	1,000
X1-5	8.17	1,220	1,040
A1-2	9.63	6.99	10.3
A1-4	9.94	(24.9) (24.8)	(83.1) (82.2)
N2-2	8.11	16.2	38.2
N2-5	8.07	15.6	31.8
X2-2	8.01	1,230	1,140
X2-5	7.99	573	673
A2-2	(9.77) (9.65)	23.2	62.3
A2-2 EXT. DUP	N/A	24.2	66.3
A2-4	9.65	21.1	57.4
Blank	N/A	<.5	<.5
WPO	N/A	11.5	60.9
WPO T.V.	N/A	10.8	58.0
Check Std.	6.96	5.55	5.25
Check Std. T.V.	7.00	5.00	5.00
Spike Recovery	N/A	98%	97%

SAMPLE	mg NO <sub>3</sub> -(N) per Kg soil
N1-1	21.8
N1-4	13.2
X1-1	80
X1-4	72.2
A1-1	2.4
A1-4	1.9
N2-1	10.9
N2-4	(20.2) (18.8)
X2-1	34.2
X2-4	35.6
A2-1	4.8
A2-4	1.4
A2-4 EXT. DUP	1.7
Blank	<.2
WPO	10.8
WPO T.V.	12.0
Check Std.	2.5
Check Std. T.V.	2.5
Spike Recovery	90%

Results from Fifth Sampling Event  
(Day 251)

Sampling Date: 23-March-1999

R.S. Kerr Laboratory Service Request Number: SF-0-75

3/23/99

Date Received: 25MAR99

Date Extracted: 29MAR99

Date(s) Analyzed: 21JUN99 to 30JUN99

Analyzed by RWC

File Name		694BLKIS.TXT	695A11.TXT	696A12.TXT	697A13.TXT	698A14.TXT	699A15.TXT	700_15.TXT	701A21.TXT	702A22.TXT
Sample I.D.								PCB Chk Std		
PCB Congener	Lib #	Blank	A 1-1	A 1-2	A 1-3	A 1-4	A 1-5	5 ng/ul Mix 1	A 2-1	A 2-2
26-4	1	***	0.61	0.44	0.54	0.68	0.49	***	1.45	1.41
25-26	2	***	1.79	1.44	1.81	1.61	1.56	***	2.87	2.85
24-4	3	***	0.61	0.48	0.73	0.58	0.58	5.15	0.68	0.77
24-26	4	***	0.85	0.58	0.83	0.79	0.78	***	1.08	1.12
236-2	5	***	2.30	2.02	2.28	2.02	2.11	***	3.44	3.29
23-4	6	***	1.19	0.79	0.98	0.96	0.88	5.12	1.35	1.44
23-26	7	***	1.02	0.82	1.09	0.99	0.94	***	1.52	1.50
25-25	8	***	10.80	8.75	10.37	9.70	9.54	5.13	15.63	15.76
24-25	9	***	8.16	6.84	7.69	7.00	7.24	***	11.41	11.32
24-24	10	***	3.35	2.67	3.26	2.94	2.86	***	4.93	4.92
23-25	11	***	12.49	10.16	11.75	10.73	10.45	5.17	17.52	17.30
236-3	12	***	0.54	0.75	0.87	0.68	0.49	***	1.15	0.93
23-24	13	***	4.33	3.28	3.81	3.59	3.64	***	5.71	5.95
26-34	14	***	2.68	1.92	2.36	1.95	2.08	5.02	4.46	4.70
234-2	15	***	0.88	0.92	0.91	0.68	0.91	*	1.62	1.66
236-4	16	***	6.60	5.64	6.38	5.64	5.71	***	9.18	9.15
23-23	17	***	2.68	2.50	2.72	2.49	2.69	***	3.95	4.09
235-4	18	***	0.64	0.58	0.51	0.55	0.58	***	0.88	0.90
236-25	19	***	3.96	3.80	3.99	4.03	3.86	***	6.18	6.01
245-4	20	***	5.11	4.31	5.00	4.37	4.58	4.91	7.70	7.35
25-34	21	***	0.78	0.75	0.76	0.75	0.71	*	1.22	1.09
24-34	22	***	9.89	8.62	9.72	9.12	8.83	4.79	14.11	14.01
235-25	23	***	0.85	0.72	0.83	0.82	0.81	***	1.38	***
23-34	24	***	5.89	5.06	5.58	5.12	5.29	4.78	8.68	8.35
235-24 or 245-25	25	***	4.57	3.97	4.21	4.00	3.99	***	6.58	6.20
234-4	26	***	3.79	3.49	3.84	3.38	3.54	***	6.14	5.98
245-24	27	***	2.88	2.63	2.79	2.87	2.76	4.73	4.29	4.19
245-23	28	***	2.51	2.15	2.25	2.29	2.18	***	3.54	3.45
234-25	29	***	2.81	2.63	2.94	2.49	2.56	4.74	4.05	4.00
234-24	30	***	1.93	1.57	1.85	1.78	1.95	***	3.21	2.81
236-34	31	***	5.49	4.96	5.37	4.89	5.32	4.66	8.41	8.06
234-23	32	*	1.59	1.50	1.34	1.64	1.72	4.68	2.60	2.53
34-34	33	***	0.81	0.72	0.91	0.79	0.81	***	1.45	1.37
245-34	34	***	3.89	3.45	3.81	3.55	3.73	***	5.84	5.88
234-34	35	***	2.84	2.43	2.97	2.90	2.82	***	4.32	4.38
Total PCB		0.00	121.12	103.35	117.03	108.37	109.01	58.90	178.52	174.72

\*\*\* Indicates extract conc. &lt; low calib. std. (0.1 ng/ul)

\* Indicates compound was not detected in extract

Date Received: 25MAR99

Date Extracted: 29MAR99

Date(s) Analyzed: 21JUN99 to 30JUN99

Analyzed by RWC

File Name		703A23.TXT	704A24.TXT	705A25.TXT	706_25.TXT	707X11A.TXT	708X11B.TXT	709X12A.TXT	710X12B.TXT	711X13.TXT
Sample I.D.					PCB Chk Std					
PCB Congener	Lib #	A 2-3	A 2-4	A 2-5	5 ng/ul Mix 2	X 1-1a	X 1-1b	X 1-2a	X 1-2b	X 1-3
26-4	1	1.57	1.34	1.38	5.10	***	***	***	***	0.23
25-26	2	3.13	3.03	2.91	***	0.52	***	***	0.49	0.47
24-4	3	0.69	0.63	0.96	*	0.83	0.71	0.70	0.69	0.77
24-26	4	1.17	1.02	1.03	***	***	***	***	***	***
236-2	5	3.42	3.17	3.58	4.88	***	0.68	0.49	0.59	0.43
23-4	6	1.38	1.27	1.63	***	***	***	***	0.36	0.33
23-26	7	1.78	1.51	1.60	4.86	***	***	***	***	***
25-25	8	15.66	15.13	15.49	***	2.52	2.44	2.50	2.44	2.30
24-25	9	11.58	10.98	11.13	***	1.87	1.98	1.90	1.88	1.87
24-24	10	4.84	4.47	4.75	***	0.80	0.84	0.84	0.72	0.84
23-25	11	18.10	16.53	16.91	***	2.87	2.66	2.85	2.60	2.57
236-3	12	1.20	1.30	0.92	***	0.10	0.19	0.14	0.10	0.13
23-24	13	5.68	5.28	5.96	***	1.00	0.91	0.95	0.82	0.84
26-34	14	4.66	4.54	4.54	*	0.93	0.97	1.06	0.72	0.77
234-2	15	1.35	1.41	1.52	4.68	0.38	0.42	0.28	0.30	0.30
236-4	16	9.47	8.86	9.04	***	1.76	1.66	1.69	1.58	1.57
23-23	17	4.19	3.98	3.90	***	0.90	0.68	0.92	0.76	0.57
235-4	18	0.95	0.88	0.92	***	***	***	***	***	***
236-25	19	6.63	5.31	5.99	4.61	1.18	1.10	1.44	1.09	1.14
245-4	20	7.25	6.93	7.55	*	1.42	1.36	1.55	1.42	1.17
25-34	21	1.09	0.91	1.45	4.54	0.83	0.84	1.27	0.76	0.60
24-34	22	14.68	13.09	14.11	***	2.84	2.99	3.41	2.73	2.44
235-25	23	1.35	1.13	1.24	*	0.45	0.32	0.42	***	0.20
23-34	24	8.96	7.99	8.47	***	1.59	1.66	1.90	1.51	1.37
235-24 or 245-25	25	6.56	5.95	6.35	***	1.42	1.43	1.62	1.28	1.10
234-4	26	6.23	5.52	5.74	4.40	1.04	1.04	1.16	1.02	1.00
245-24	27	4.30	3.90	4.29	***	0.93	0.88	1.06	0.82	0.80
245-23	28	3.93	3.38	3.47	***	0.73	0.71	0.99	0.72	0.70
234-25	29	4.37	3.48	3.86	***	1.00	0.84	1.13	0.86	0.80
234-24	30	3.46	2.85	2.94	*	0.52	0.55	0.70	0.59	0.50
236-34	31	8.71	7.49	7.83	***	1.59	1.59	2.01	1.68	1.44
234-23	32	2.55	2.39	2.27	***	***	***	0.63	***	0.43
34-34	33	1.38	1.34	1.49	*	0.28	0.23	0.42	0.30	***
245-34	34	5.97	5.28	5.57	***	1.21	1.20	1.37	1.15	1.14
234-34	35	4.48	3.87	4.29	***	0.93	0.94	1.02	0.92	0.84
Total PCB		182.75	166.11	175.09	33.12	32.44	31.88	36.43	30.90	29.66

\*\*\* Indicates extract conc. &lt; low calib. std. (0.1 ng/ul)

\* indicates compound was not detected in extract

Date Received: 25MAR99

Date Extracted: 29MAR99

Date(s) Analyzed: 21JUN99 to 30JUN99

Analyzed by RWC

File Name		712_35.TXT	713X14.TXT	714X15A.TXT	715X15B.TXT	716X21.TXT	717X22.TXT	718_45.TXT	719X23.TXT	720X24A.TXT
Sample I.D.		PCB Chk Std						PCB Chk Std		
PCB Congener	Lib #	5 ng/ul Mix 3	X 1-4	X 1-5a	X 1-5b	X 2-1	X 2-2	5 ng/ul Mix 4	X 2-3	X 2-4a
26-4	1	***	***	***	***	0.23	***	***	0.23	***
25-26	2	***	0.41	***	***	0.43	0.51	3.99	0.43	0.47
24-4	3	***	0.66	0.72	0.71	0.67	0.66	***	0.74	0.73
24-26	4	***	***	***	***	***	***	3.90	***	***
236-2	5	***	0.44	0.56	0.54	0.50	***	***	***	0.63
23-4	6	***	0.32	0.36	***	0.33	0.25	***	0.30	***
23-26	7	***	***	***	***	0.30	***	*	***	***
25-25	8	*	2.11	2.43	2.11	2.23	1.88	***	2.31	2.33
24-25	9	***	1.73	2.13	1.70	1.83	1.52	***	1.70	1.87
24-24	10	4.79	0.79	0.92	0.89	0.77	0.86	*	0.84	0.73
23-25	11	***	2.37	2.69	2.55	2.43	2.39	***	2.57	2.60
236-3	12	***	0.09	0.16	0.10	0.10	0.10	***	0.07	0.20
23-24	13	4.87	0.98	1.08	0.92	1.00	0.81	***	0.90	0.97
26-34	14	***	0.76	0.92	0.85	0.77	0.81	***	0.84	0.87
234-2	15	***	0.25	0.49	0.34	***	0.30	***	0.33	0.27
236-4	16	*	1.48	1.61	1.50	1.27	1.32	*	1.57	1.53
23-23	17	4.86	0.60	0.85	0.85	0.63	0.86	*	0.67	0.63
235-4	18	***	***	***	***	***	***	***	***	***
236-25	19	*	1.01	1.18	1.23	1.07	1.07	***	1.07	1.10
245-4	20	***	1.20	1.48	1.43	1.27	1.37	***	1.30	1.30
25-34	21	***	0.69	0.75	0.71	0.67	0.76	***	0.70	0.73
24-34	22	***	2.40	2.85	2.55	2.33	2.29	***	2.57	2.57
235-25	23	4.75	***	0.39	***	***	***	***	***	0.27
23-34	24	*	1.42	1.61	1.57	1.33	1.47	***	1.47	1.47
235-24 or 245-25	25	4.22	1.10	1.31	1.29	1.03	1.27	3.82	1.27	1.27
234-4	26	***	0.82	1.05	0.95	0.90	0.86	***	0.90	0.93
245-24	27	***	0.69	0.85	0.85	0.77	***	***	0.74	0.80
245-23	28	***	0.66	0.89	0.75	0.60	***	***	0.80	0.73
234-25	29	***	0.69	0.89	0.75	0.77	0.81	*	0.74	0.77
234-24	30	***	0.57	0.56	0.51	0.53	0.30	*	0.50	0.53
236-34	31	***	1.36	1.67	1.60	1.37	1.47	***	1.54	1.50
234-23	32	***	0.50	0.62	0.48	***	***	***	0.57	0.53
34-34	33	*	0.32	***	0.27	0.30	***	***	0.27	0.33
245-34	34	4.68	1.01	1.25	1.26	1.03	1.07	***	1.10	1.10
234-34	35	4.71	0.95	0.85	0.92	0.90	1.02	***	0.94	1.00
Total PCB		32.91	28.39	33.14	30.20	28.37	26.07	11.79	29.97	30.75

\*\*\* indicates extract conc. &lt; low calib. std. (0.1 ng/ul)

\* indicates compound was not detected in extract

Date Received: 25MAR99

Date Extracted: 29MAR99

Date(s) Analyzed: 21JUN99 to 30JUN99

Analyzed by RWC

File Name		721X24B.TXT	722X25.TXT	723N11.TXT	724_55.TXT	725N12.TXT	726N13.TXT	727N14.TXT	728N15.TXT	729N21.TXT
Sample I.D.					PCB Chk Std					
PCB Congener	Lib #	X 2-4b	X 2-5	N 1-1	5 ng/ul Mix 5	N 1-2	N 1-3	N 1-4	N 1-5	N 2-1
26-4	1	***	***	***	***	***	***	***	***	***
25-26	2	0.58	0.54	0.73	***	0.76	0.97	0.96	***	0.74
24-4	3	0.77	0.68	1.46	*	1.33	1.41	1.49	1.13	1.19
24-26	4	***	***	***	***	***	***	***	***	***
236-2	5	0.58	0.72	1.00	***	0.79	***	1.06	***	***
23-4	6	0.32	0.36	0.67	***	0.65	0.74	0.81	0.67	0.56
23-26	7	***	***	***	***	***	***	***	***	***
25-25	8	2.54	2.44	4.72	***	4.44	4.43	5.21	4.40	3.79
24-25	9	2.00	1.94	3.89	4.27	3.68	3.59	4.07	3.63	2.94
24-24	10	0.93	0.90	1.70	***	1.59	1.48	1.81	1.51	1.45
23-25	11	2.93	2.66	5.52	***	5.05	5.07	5.95	4.86	4.16
236-3	12	0.19	0.27	0.47	4.27	0.32	0.27	0.25	0.39	0.33
23-24	13	0.93	1.17	1.73	*	1.80	1.75	2.20	1.94	1.75
26-34	14	0.97	0.86	1.26	***	1.19	1.01	1.52	1.20	0.97
234-2	15	***	***	0.57	***	***	0.50	0.78	0.56	***
236-4	16	1.58	1.71	3.29	4.33	2.96	3.06	3.22	3.03	2.53
23-23	17	0.77	0.77	1.23	*	1.37	1.44	1.81	1.51	1.30
235-4	18	0.13	***	***	4.26	0.40	0.37	***	***	0.45
236-25	19	1.06	1.17	2.29	***	2.16	2.32	2.59	1.87	1.78
245-4	20	1.38	1.31	2.83	***	2.45	2.69	3.36	2.53	2.45
25-34	21	0.93	0.86	1.46	***	1.12	1.24	1.56	1.02	1.30
24-34	22	2.90	2.89	5.95	***	5.12	5.10	6.98	5.10	4.61
235-25	23	***	0.36	0.67	***	***	0.47	0.81	0.60	0.48
23-34	24	1.54	1.67	3.46	***	2.92	2.92	4.11	2.85	2.60
235-24 or 245-25	25	1.35	1.44	2.63	***	2.45	2.12	3.40	2.32	2.04
234-4	26	1.03	1.08	2.26	***	1.95	1.85	2.62	1.97	1.71
245-24	27	0.87	0.90	1.73	***	1.59	1.54	2.23	1.58	1.52
245-23	28	0.90	***	1.53	4.23	1.52	1.41	1.84	1.27	1.38
234-25	29	0.87	0.77	1.76	***	1.55	1.51	2.05	1.65	1.52
234-24	30	0.61	0.45	1.26	4.15	1.15	0.97	1.56	1.30	1.12
236-34	31	1.74	1.53	3.36	***	3.03	2.92	4.43	2.89	2.79
234-23	32	***	0.68	1.06	***	***	0.97	1.31	1.16	***
34-34	33	0.35	0.36	0.60	4.67	0.69	0.64	0.89	***	***
245-34	34	1.29	1.40	2.46	***	2.27	2.12	3.26	2.08	2.19
234-34	35	1.09	0.95	1.90	***	1.80	1.65	2.62	1.94	1.67
Total PCB		33.14	32.83	65.44	30.25	58.12	58.52	76.74	56.96	51.35

\*\*\* indicates extract conc. &lt; low calib. std. (0.1 ng/ul)

\* indicates compound was not detected in extract

Date Received: 25MAR99

Date Extracted: 29MAR99

Date(s) Analyzed: 21JUN99 to 30JUN99

Analyzed by RWC

File Name		730_1P5.TXT	731N22.TXT	732N22S.TXT	733N23.TXT	734N23S.TXT	735N24.TXT	736_2P5.TXT	737N25.TXT	738XBLK.TXT
Sample I.D.		PCB Chk Std						PCB Chk Std		Extraction
PCB Congener	Lib #	0.5 ng/ul Mix 1	N 2-2	N 2-2 small	N 2-3	N 2-3 small	N 2-4	0.5 ng/ul Mix 2	N 2-5	Blank #1
26-4	1	***	***	***	***	***	***	0.56	***	***
25-28	2	***	***	0.68	0.76	0.74	0.57	***	0.61	***
24-4	3	0.52	1.17	1.11	1.21	1.51	1.04	*	1.08	***
24-26	4	***	***	***	***	***	***	***	***	***
236-2	5	***	0.77	0.90	1.02	1.07	***	0.48	0.79	***
23-4	6	0.50	0.50	0.58	0.57	0.78	0.47	***	0.47	***
23-26	7	***	***	***	***	***	***	0.52	***	***
25-25	8	0.45	3.80	3.74	4.21	4.84	3.26	*	3.43	***
24-25	9	***	2.90	2.88	3.34	3.58	2.37	***	2.75	***
24-24	10	***	1.43	1.26	1.40	1.55	1.14	***	1.41	***
23-25	11	0.53	4.14	4.13	4.59	5.35	3.41	***	3.83	***
236-3	12	***	0.27	0.36	0.30	0.30	0.90	***	0.07	***
23-24	13	***	1.73	1.40	1.93	1.92	1.28	***	1.59	***
26-34	14	0.51	0.80	0.86	1.29	1.26	0.90	*	0.83	***
234-2	15	***	***	***	0.57	0.59	***	0.45	***	***
236-4	16	***	2.37	2.37	2.88	3.07	2.08	***	2.20	***
23-23	17	***	1.07	1.15	1.44	1.29	1.04	***	1.23	***
235-4	18	*	0.33	0.29	0.38	0.37	0.38	***	0.29	***
236-25	19	***	1.80	1.91	2.20	2.22	1.61	0.52	1.73	***
245-4	20	0.50	2.17	2.26	2.62	2.73	1.94	*	2.13	***
25-34	21	*	1.20	1.33	1.55	1.59	1.14	0.43	1.23	***
24-34	22	0.53	4.34	4.57	5.23	5.65	4.26	***	4.34	***
235-25	23	***	0.47	0.40	0.61	0.70	0.43	*	0.58	***
23-34	24	0.50	2.43	2.48	2.99	2.99	2.41	*	2.35	***
235-24 or 245-25	25	***	1.93	2.05	2.35	2.58	1.85	***	1.99	***
234-4	26	***	1.67	1.80	1.97	2.10	1.51	0.47	1.55	***
245-24	27	0.54	1.43	1.26	1.55	1.66	1.37	***	1.37	***
245-23	28	***	1.23	1.08	1.33	1.48	1.04	***	1.16	***
234-25	29	0.49	1.33	1.37	1.67	1.48	1.32	*	1.23	***
234-24	30	***	0.87	0.86	1.02	1.14	0.80	***	0.87	***
236-34	31	0.50	2.53	2.52	2.96	3.03	2.37	***	2.42	***
234-23	32	0.51	0.83	0.79	0.99	1.11	0.99	***	0.90	***
34-34	33	***	0.40	0.47	0.61	0.74	0.57	***	0.51	***
245-34	34	***	1.80	1.91	2.24	2.25	1.89	***	1.95	***
234-34	35	***	1.40	1.55	1.90	1.81	1.42	***	1.48	***
Total PCB		6.10	49.13	50.30	59.67	63.48	45.75	3.51	48.37	0.00

\*\*\* Indicates extract conc. &lt; low calib. std. (0.1 ng/ul)

\* Indicates compound was not detected in extract

Date Received: 25MAR99

Date Extracted: 29MAR99

Date(s) Analyzed: 21JUN99 to 30JUN99

Analyzed by RWC

File Name		739A11.TXT	740A26.TXT	741X11.TXT	742_3P5.TXT	743N21.TXT	744XBLK.TXT			
Sample I.D.		SF-0-1	SF-0-1	SF-0-1	PCB Chk Std	SF-0-1	Extraction			
PCB Congener	Lib #	A 1-1	A 2-6	X 1-1	0.5 ng/ul Mix 3	N 2-1	Blank #2			
26-4	1	1.25	1.28	***	*	0.92	*			
25-26	2	2.10	2.20	0.49	***	1.33	***			
24-4	3	0.91	0.81	0.54	*	2.12	***			
24-26	4	0.88	1.08	***	***	***	***			
236-2	5	2.20	2.33	***	***	1.30	***			
23-4	6	1.29	1.45	*	***	1.01	***			
23-26	7	1.15	1.22	***	***	0.73	***			
25-25	8	10.53	11.63	1.74	***	6.33	***			
24-25	9	7.62	8.62	1.38	***	4.59	***			
24-24	10	3.08	3.45	***	0.50	2.03	***			
23-25	11	11.10	12.81	1.87	*	6.27	***			
236-3	12	0.41	0.78	*	***	0.41	***			
23-24	13	4.10	4.36	*	0.51	2.31	***			
26-34	14	3.69	4.12	0.68	***	2.03	***			
234-2	15	1.22	1.59	***	***	1.08	***			
236-4	16	6.13	7.03	1.00	***	3.67	***			
23-23	17	2.71	3.24	***	0.48	1.65	***			
235-4	18	0.71	0.81	0.08	***	0.54	***			
236-25	19	4.06	4.80	0.87	***	2.72	***			
245-4	20	5.65	6.15	0.95	***	3.48	***			
25-34	21	3.55	2.97	0.60	***	2.12	***			
24-34	22	10.60	12.40	1.76	***	6.65	***			
235-25	23	0.88	1.22	***	0.48	0.57	***			
23-34	24	5.92	7.03	1.11	***	3.64	***			
235-24 or 245-25	25	4.30	5.00	0.87	0.46	2.91	***			
234-4	26	4.06	4.60	0.73	***	2.60	***			
245-24	27	2.95	3.38	0.60	***	2.12	***			
245-23	28	2.47	2.91	0.57	***	1.55	***			
234-25	29	2.81	3.31	0.65	***	1.96	***			
234-24	30	2.27	2.67	0.49	***	1.39	***			
236-34	31	5.45	6.39	1.11	***	3.42	***			
234-23	32	1.73	2.13	***	***	1.14	***			
34-34	33	0.98	1.22	***	*	0.76	***			
245-34	34	3.93	4.66	0.79	0.44	2.57	***			
234-34	35	2.98	3.62	0.62	0.45	1.77	***			
Total PCB		125.66	143.27	19.51	3.35	79.72	0.00			

\*\*\* indicates extract conc. &lt; low calib. std. (0.1 ng/ul)

\* indicates compound was not detected in extract

Sampling Event #5  
3/23/99



• **MEMORANDUM**  
**MANTECH ENVIRONMENTAL RESEARCH SERVICES CORP.**  
Environmental Science

In reply refer to :99-KPJ2  
Contract # 68-C-98-138

To: Dr. Kampbell

From: Ken Jewell

Thru: D.D. Fine *fine*

Date: 04-05-99

Subject: Report letter for SF-0-75

Copies: R.L. Cosby,  
J.L. Seeley *JS*  
G.B. Smith

Ref: 99-KPJ-2

As per service request SF-0-75, the soil samples from Cape Canaveral have been extracted for GCMS analysis and percent moisture has been calculated. For percent moisture calculation, ten grams of soil were used and the following equation was applied:

(Wet soil- Dry soil)/(Wet soil)X100

Sample#	%Moisture
A1-1	11.20
A1-2	10.87
A1-3	10.69
A1-4	9.44
A1-5	14.35
A2-1	9.47
A2-2	9.73
A2-3	7.60
A2-4	10.75
A2-5	8.84
X1-1	13.55
X1-1b	12.72
X1-2	10.66
X1-2b	10.77
X1-3	12.94
X1-4	13.86
X1-5	12.56
X1-5b	10.83
X2-1	12.59

ManTech Environmental Research Services Corporation

R.S. Kerr Environmental Research Laboratory, P.O. Box 1198, 919 Research Drive  
Ada, Oklahoma 74821-1189 405-436-8660 FAX 405 436 8501

Sample I.D.	% Moisture
X2-2	13.51
X2-3	13.74
X2-4	14.34
X2-4b	14.19
X2-5	13.13
N1-1	5.57
N1-2	4.50
N1-3	5.46
N1-4	4.57
N1-5	5.42
N2-1	3.33
N2-2	3.14
N2-2 Small Core	2.29
N2-3	2.59
N2-3 Small Core	1.96
N2-4	1.54
N2-5	4.90

If you have any questions concerning the information contained in this report, please contact me at your convenience.

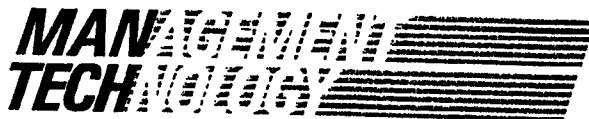


Ken Jewell

xc: R. Cosby  
J. Seeley  
D. Fine

S.E. #5

3/23/99



MEMORANDUM

MANTECH ENVIRONMENTAL RESEARCH SERVICES CORP.  
Environmental Science

In reply refer to: 99-PR28  
Contract # 68-C-98-138

To: Dr. Don Kampbell

From: Priscilla Rodebush PR

Thru: Dr. Dennis Fine *dfine*

Subject: SF-0-75

Date: April 8, 1999

Copies: J.L. Seeley *JS*  
R.L. Cosby  
G.B. Smith

Attached is the report for the HPLC analysis of acetic acid and butyric acid for the samples received March 25, 1999. The samples were analyzed using a Waters 431 Conductivity detector in units of  $\mu\text{M}$ . Check standards (CS 120 $\mu\text{M}$ ) and standards (STD 200 $\mu\text{M}$ ) were used in the analysis. The sample injection volume was 50  $\mu\text{l}$  with a calibration range of 10 $\mu\text{M}$  to 600 $\mu\text{M}$ .

The column and eluent were a Dionex ICE-AS1 IonPac column with 1.0 mM heptafluorobutyric acid, 10% acetonitrile and 90% DI water. An AMMS-ICE MicroMembrane Suppressor with 5 mM tetrabutylammonium hydroxide reagent was used for all analyses. The flow rate was 0.8 ml/min for the eluent and 5.0 ml/min for the suppressor reagent.

The extraction procedure is as follows: weigh 3 grams of soil into a flask, add 100 mL of 0.1 M HCl to each flask. Place the samples on a shaker with a rotation rate of 150 cycles per minute for 24 hours. Filter the extract through 0.2  $\mu\text{M}$  syringe filters. The extractions were performed by Sean Beach.

Acetic and butyric acid were not detected in any of the samples analyzed, therefore the results are reported in  $\mu\text{M}$  and not  $\mu\text{M/kg}$ . These samples were analyzed on April 5 and 6, 1999. This analysis was performed by Priscilla Rodebush.

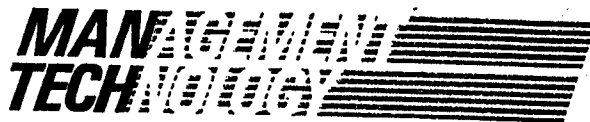
ManTech Environmental Research Services Corporation

R.S. Kerr Environmental Research Center, P.O. Box 1198, 919 Kerr Research Drive  
Ada, Oklahoma 74821-1198 580-436-8660 FAX 580-436-8501

Service Request: SF-0-75  
Originator: Dr. D. Kampbell  
Date Received: 3/25/99  
Date Analyzed: 4/5/99  
Analyst: Priscilla Rodebush

SampleName	acetic acid (uM)	butyric acid (uM)
CS 120 uM	112	132
STD 200 uM	206	228
HPLC BLANK	ND	ND
A1 3	ND	ND
A1 5	ND	ND
A2 3	ND	ND
A2 5	ND	ND
N1 3	ND	ND
N2 3	ND	ND
X1 3	ND	ND
X2 3	ND	ND
BLANK	ND	ND
A2 5 dup	ND	ND
HPLC BLANK	ND	ND
CS 120 uM	114	133
STD 200 uM	211	224

ND =non detected



**MEMORANDUM**

**MANTECH ENVIRONMENTAL RESEARCH SERVICES CORP.**  
**Environmental Science**

In reply refer to: 99-37LP/lp  
Contract # 68-C-98-138

To: Dr. Don Kampbell

Thru: D.D. Fine *DDF*

From: Lynda Pennington *LKP*

Subject: SR # SF-0-75

Ref:

Copies: R.L. Cosby  
G.B. Smith  
J.L. Seeley *JLS*

Date: April 29, 1999

Attached are inorganic results for 24 Cape Canaveral, Florida soil samples submitted to MERSC under Service Request # SF-0-75. The samples were received March 25, stored in the refrigerator until a portion was removed from each sample and air dried on April 20, and were analyzed April 26 and 27, 1999. The soils were extracted 1:1 with RO water for pH, chloride and sulfate and 1:2 with 2 M potassium chloride for nitrate analysis. The methods used were Waters capillary electrophoresis method N-601 for chloride and sulfate; Lachat FIA methods 10-107-04-2-A for nitrate; and EPA method 150.1 for pH.

Quality control measures performed along with your samples included analysis of blanks, duplicates, spikes, known WPO samples and check standards.

If you have any questions concerning this data, please feel free to contact me.

ManTech Environmental Research Services Corporation

R.S. Kerr Environmental Research Center, P.O. Box 1198, 919 Kerr Research Drive  
Ada, Oklahoma 74821-1198 580.436.8660 FAX 580.436.9501

Samples Rec'd 3-25-99  
 Subsampled 4-20-99  
 Analyzed 4-26 and 4-27-99  
 by L. Pennington

S.R. # SF-0-75  
 Don Kampbell  
 Cape Canaveral, FL soils

Page 1

SAMPLE	pH	Chloride mg/Kg soil	Sulfate mg/Kg soil	SAMPLE	Nitrate mg/Kg soil
N1-2	8.06	21.9	42	N1-1	16.4
N1-5	8.12	22.1	38.8	N1-4	14.6
X1-2	8.08	1,460	1,060	X1-1	94.8
X1-5	8.11	1,530	1,460	X1-4	(67.5) (68.1)
A1-2	10.2	16.3	55.4	A1-1	5.58
A1-4	10.3	(15.8) (15.8)	(66.3) (66.1)	A1-4	6.29
N2-2	(8.08) (8.08)	(17.1) (17.2)	(33.0) (33.0)	N2-1	18.3
N2-5	8.05	18.8	26.2	N2-4	12.9
X2-2	8.13	1,030	1,130	X2-1	52.2
X2-5	8.11	1,990	1,860	X2-4	62.1
A2-2	10.2	22.2	60.4	A2-1	6.17
A2-4	10.1	18.0	68.1	A2-4	(5.67) (6.02)
Blank	N/A	<1.00	<1.00	Blank	<1.00
AQC	N/A	11.3	62.8	AQC	N/A
AQC T.V.	N/A	10.8	58.0	AQC T.V.	N/A
Check Std.	7.00	5.14	5.23	Check Std.	4.75
Check Std. T.V.	7.00	5.00	5.00	Check Std. T.V.	5.00
Spike Recovery	N/A	99%	100%	Spike Recovery	88%

Results from Sixth Sampling Event  
(Day 476)

Sampling Date: 3-November-1999

R.S. Kerr Laboratory Service Request Number: SF-0-151

Analyte Concentrations are  $\mu\text{g}$  compound / g soil, unless otherwise indicated

File Name:		046BLKIS.TXT	047_11.TXT	048A11.TXT	049A12.TXT	050A13.TXT	051A14.TXT	052A15.TXT	053_21.TXT	054A21.TXT
Sample ID:		Blank Isooctane	PCB Cngr Chk Std	A 1-1	A 1-2	A 1-3	A 1-4	A 1-5	PCB Cngr Chk Std	A 2-1
Analyte	Libr. #	(diluent)	1.0 ng/ $\mu\text{l}$ mix #1						1.0 ng/ $\mu\text{l}$ mix #2	
biphenyl	1	*	*	9.41	5.63	7.39	9.24	8.91	*	7.39
26-4	2	*	*	0.03	0.06	0.04	0.02	0.07	1.17	0.13
25-26	3	*	*	0.12	0.14	0.12	0.07	0.13	*	0.30
24-4	4	*	1.11	0.05	0.06	0.05	0.03	0.07	*	0.08
24-26	5	*	*	0.04	0.05	0.04	0.03	0.05	*	0.10
236-2	6	*	*	0.17	0.20	0.18	0.12	0.18	1.16	0.42
23-4	7	*	1.17	0.08	0.09	0.08	0.05	0.08	*	0.16
23-26	8	*	*	0.07	0.08	0.07	0.05	0.08	1.15	0.16
25-25	9	*	1.21	0.87	1.06	0.86	0.55	0.87	*	2.14
24-25	10	*	*	0.69	0.80	0.67	0.38	0.66	*	1.56
24-24	11	*	*	0.26	0.28	0.25	0.14	0.24	*	0.53
23-25	12	*	1.15	1.14	1.12	0.97	0.64	0.93	*	2.46
236-3	13	*	*	0.08	0.06	0.07	0.03	0.05	*	0.14
23-24	14	*	*	0.27	0.32	0.25	0.17	0.26	*	0.69
26-34	15	*	1.16	0.18	0.24	0.17	0.11	0.19	*	0.55
234-2	16	*	*	0.06	0.07	0.05	0.04	0.05	1.22	0.15
236-4	17	*	*	0.61	0.59	0.53	0.32	0.52	*	1.24
23-23	18	*	*	0.20	0.21	0.19	0.12	0.19	*	0.47
235-4	19	*	*	0.05	0.06	0.05	0.03	0.05	*	0.13
236-25	20	*	*	0.38	0.38	0.34	0.21	0.32	1.18	0.89
245-4	21	*	1.14	0.42	0.44	0.42	0.24	0.41	*	0.99
25-34	22	*	*	0.05	0.06	0.05	0.03	0.05	1.15	0.11
24-34	23	*	1.18	0.87	0.93	0.82	0.50	0.81	*	2.03
235-25	24	*	*	0.08	0.08	0.06	0.04	0.06	*	0.15
23-34	25	*	1.20	0.52	0.55	0.50	0.30	0.48	*	1.27
235-24 and 245-25	26	*	*	0.36	0.38	0.35	0.21	0.31	*	0.89
234-4	27	*	*	0.38	0.37	0.31	0.20	0.31	1.09	0.85
245-24	28	*	1.15	0.27	0.27	0.23	0.14	0.24	*	0.60
245-23	29	*	*	0.22	0.23	0.20	0.13	0.20	*	0.49
234-25	30	*	1.14	0.26	0.26	0.23	0.15	0.22	*	0.57
234-24	31	*	*	0.18	0.16	0.17	0.09	0.15	*	0.41
236-34	32	*	1.08	0.51	0.52	0.42	0.28	0.43	*	1.21
234-23	33	*	1.13	0.16	0.15	0.14	0.09	0.12	*	0.34
34-34	34	*	*	0.10	0.09	0.08	0.05	0.08	*	0.20
245-34	35	*	*	0.37	0.35	0.32	0.20	0.30	*	0.77
234-34	36	*	*	0.27	0.27	0.24	0.17	0.22	*	0.64
Total Mass of Quantified PCB Congeners (mg/Kg)		NA	NA	10.39	10.97	9.53	5.93	9.38	NA	23.81

\* analyte not detected in extract

\*\*\* conc. of analyte in extract < 0.05 ng/ $\mu\text{l}$ 

biphenyl concentrations are ESTIMATES ONLY

NA = not applicable

Analyte Concentrations are µg compound / g soil, unless otherwise indicated

File Name:		055A22.TXT	056A22D.TXT	057A23.TXT	058A23D.TXT	059_31.TXT	060A24.TXT	061A25.TXT	062X11.TXT	063X12.TXT
Sample ID:		A 2-2	A 2-2 dup	A 2-3	A 2-3 dup	PCB Cngr Chk Std 1.0 ng/µl mix #3	A 2-4	A 2-5	X 1-1	X 1-2
Analyte	Libr. #									
biphenyl	1	0.15	18.0	0.05	12.5	*	10.6	1.91	*	***
26-4	2	*	0.20	0.18	0.15	*	0.13	0.11	***	0.02
25-26	3	***	0.37	0.39	0.40	*	0.31	0.30	0.06	0.06
24-4	4	***	0.18	0.30	0.11	*	0.08	0.08	0.10	0.08
24-26	5	***	0.12	0.11	0.11	*	0.09	0.09	0.03	0.02
236-2	6	0.05	0.48	0.40	0.44	*	0.36	0.36	0.08	0.07
23-4	7	***	0.23	0.23	0.19	*	0.14	0.15	0.05	0.04
23-26	8	***	0.20	0.16	0.19	*	0.17	0.14	0.04	0.03
25-25	9	0.15	2.28	2.22	2.36	*	1.92	1.81	0.37	0.33
24-25	10	0.11	1.80	1.74	1.71	*	1.40	1.40	0.28	0.24
24-24	11	0.05	0.61	0.68	0.63	1.12	0.52	0.49	0.11	0.09
23-25	12	0.16	2.83	2.78	2.80	*	2.21	2.22	0.43	0.37
236-3	13	***	0.13	0.18	0.14	*	0.09	0.15	0.03	0.03
23-24	14	0.07	0.79	0.74	0.79	1.14	0.66	0.61	0.13	0.11
26-34	15	0.06	0.75	0.69	0.71	*	0.53	0.55	0.13	0.12
234-2	16	*	0.17	0.18	0.17	*	0.13	0.15	0.03	0.03
236-4	17	0.10	1.39	1.40	1.42	*	1.20	1.18	0.23	0.21
23-23	18	0.04	0.51	0.42	0.44	1.00	0.37	0.38	0.08	0.07
235-4	19	***	0.14	0.14	0.14	*	0.10	0.11	0.04	0.03
236-25	20	0.04	0.99	1.09	1.02	*	0.84	0.89	0.18	0.15
245-4	21	0.09	1.17	1.14	1.06	*	0.85	0.87	0.22	0.19
25-34	22	***	0.17	0.40	0.14	*	0.12	0.11	0.10	0.09
24-34	23	0.16	2.37	2.28	2.26	*	1.99	1.84	0.40	0.39
235-25	24	***	0.17	0.19	0.20	1.11	0.15	0.15	0.04	0.04
23-34	25	0.08	1.41	1.40	1.43	*	1.13	1.19	0.26	0.22
235-24 and 245-25	26	0.06	0.94	1.06	0.97	1.18	0.84	0.85	0.19	0.17
234-4	27	0.06	0.94	0.90	0.90	*	0.73	0.81	0.15	0.15
245-24	28	0.06	0.68	0.75	0.70	*	0.58	0.62	0.13	0.13
245-23	29	0.04	0.55	0.61	0.58	*	0.50	0.48	0.11	0.11
234-25	30	0.05	0.66	0.69	0.71	*	0.60	0.59	0.13	0.11
234-24	31	0.04	0.39	0.43	0.42	*	0.39	0.34	0.10	0.09
236-34	32	0.09	1.25	1.37	1.34	*	1.13	1.14	0.24	0.22
234-23	33	0.06	0.37	0.39	0.38	*	0.33	0.33	0.09	0.08
34-34	34	0.04	0.22	0.24	0.21	*	0.18	0.21	0.06	0.06
245-34	35	0.08	0.90	0.94	0.85	1.14	0.80	0.84	0.18	0.17
234-34	36	0.05	0.74	0.76	0.74	1.36	0.63	0.69	0.14	0.14
Total Mass of Quantified PCB Congeners (mg/Kg)		1.79	27.09	27.62	26.79	NA	22.23	22.26	4.94	4.48

\* analyte not detected in extract

\*\*\* conc. of analyte in extract &lt; 0.05 ng/µl

biphenyl concentrations are *ESTIMATES ONLY*

NA = not applicable

Analyte Concentrations are µg compound / g soil, unless otherwise indicated

File Name:		064X12D.TXT	065_41.TXT	066X13.TXT	067X14.TXT	068X15.TXT	069X21.TXT	070X22.TXT	071_51.TXT	072X14D.TXT
Sample ID:		X 1-2 dup	PCB Cngr Chk Std 1.0 ng/µl mix #4	X 1-3	X 1-4	X 1-5	X 2-1	X 2-X	PCB Cngr Chk Std 1.0 ng/µl mix #5	X 1-4 lab dup
Analyte	Libr. #									
biphenyl	1	*	*	*	*	*	*	*	*	*
26-4	2	0.02	*	0.03	***	0.02	0.03	***	*	***
25-26	3	0.05	1.09	0.07	0.05	0.06	0.07	0.06	*	0.05
24-4	4	0.09	*	0.12	0.07	0.09	0.11	0.10	*	0.07
24-26	5	0.02	1.06	0.04	0.02	0.03	0.03	0.03	*	0.02
236-2	6	0.06	*	0.09	0.05	0.07	0.08	0.07	*	0.06
23-4	7	0.04	*	0.05	0.04	0.04	0.05	0.04	*	0.04
23-26	8	0.03	*	0.04	0.02	0.03	0.03	0.04	*	0.03
25-25	9	0.33	*	0.46	0.28	0.35	0.41	0.34	*	0.27
24-25	10	0.26	*	0.32	0.19	0.23	0.29	0.28	1.14	0.20
24-24	11	0.09	*	0.12	0.08	0.10	0.11	0.11	*	0.08
23-25	12	0.40	*	0.50	0.30	0.42	0.45	0.43	*	0.31
236-3	13	0.02	*	***	0.02	0.02	0.03	0.03	1.17	0.02
23-24	14	0.12	*	0.16	0.10	0.11	0.13	0.13	*	0.10
26-34	15	0.12	*	0.15	0.10	0.12	0.13	0.13	*	0.11
234-2	16	0.03	*	0.04	0.03	0.03	0.03	0.03	*	0.02
236-4	17	0.21	*	0.27	0.17	0.22	0.26	0.23	1.13	0.19
23-23	18	0.08	*	0.10	0.06	0.08	0.10	0.08	*	0.07
235-4	19	0.03	*	0.04	0.03	0.03	0.04	0.04	1.03	0.03
236-25	20	0.15	*	0.21	0.13	0.16	0.20	0.17	*	0.13
245-4	21	0.19	*	0.24	0.15	0.19	0.25	0.19	*	0.15
25-34	22	0.10	*	0.13	0.08	0.12	0.12	0.11	*	0.08
24-34	23	0.38	*	0.51	0.31	0.39	0.49	0.39	*	0.31
235-25	24	0.03	*	0.05	0.03	0.03	0.04	0.04	*	0.03
23-34	25	0.22	*	0.27	0.19	0.23	0.28	0.23	*	0.18
235-24 and 245-25	26	0.17	1.09	0.21	0.15	0.17	0.20	0.18	*	0.14
234-4	27	0.15	*	0.18	0.11	0.15	0.18	0.16	*	0.12
245-24	28	0.13	*	0.16	0.11	0.13	0.15	0.13	*	0.10
245-23	29	0.11	*	0.14	0.09	0.11	0.13	0.12	1.09	0.09
234-25	30	0.12	*	0.15	0.10	0.12	0.15	0.13	*	0.09
234-24	31	0.09	*	0.11	0.07	0.09	0.11	0.10	1.23	0.08
236-34	32	0.21	*	0.29	0.19	0.22	0.27	0.25	*	0.18
234-23	33	0.07	*	0.10	0.07	0.08	0.10	0.08	*	0.07
34-34	34	0.06	*	0.07	0.05	0.06	0.07	0.06	1.11	0.05
245-34	35	0.17	*	0.21	0.15	0.17	0.20	0.18	*	0.15
234-34	36	0.14	*	0.17	0.11	0.14	0.16	0.15	*	0.11
Total Mass of Quantified PCB Congeners (mg/Kg)		4.49	NA	5.80	3.71	4.60	5.48	4.84	NA	3.70

\* analyte not detected in extract

\*\*\* conc. of analyte in extract &lt; 0.05 ng/µl

NA = not applicable

Analyte Concentrations are µg compound / g soil, unless otherwise indicated

File Name:		073X23.TXT	074X24.TXT	075X25.TXT	076N11.TXT	077_2P1.TXT	078N12.TXT	079N13.TXT	080N14.TXT	081N15.TXT
Sample ID:		X 2-3	X 2-4	X 2-5	N 1-1	PCB Cngr Chk Std 0.1 ng/µl mix #2	N 1-2	N 1-3	N 1-4	N 1-5
Analyte	Libr. #									
biphenyl	1	*	*	*	*	*	*	*	*	*
26-4	2	0.03	0.03	***	0.14	0.10	0.18	0.12	0.13	0.09
25-26	3	0.07	0.06	0.06	0.10	*	0.12	0.08	0.08	0.06
24-4	4	0.10	0.10	0.10	0.25	*	0.31	0.19	0.25	0.13
24-26	5	0.03	0.03	0.03	0.04	*	0.05	0.03	0.03	0.03
236-2	6	0.08	0.08	0.08	0.01	0.13	0.02	0.01	0.01	***
23-4	7	0.05	0.05	0.05	0.02	*	0.02	0.01	0.01	***
23-26	8	0.03	0.04	0.03	***	0.11	***	***	***	***
25-25	9	0.38	0.41	0.37	0.12	*	0.16	0.11	0.13	0.08
24-25	10	0.29	0.29	0.28	0.08	*	0.11	0.07	0.08	0.05
24-24	11	0.12	0.11	0.11	0.12	*	0.15	0.11	0.11	0.08
23-25	12	0.43	0.47	0.46	0.05	*	0.09	0.05	0.07	0.03
236-3	13	***	0.04	0.03	***	*	***	***	***	***
23-24	14	0.15	0.14	0.14	0.03	*	0.04	0.03	0.03	0.02
26-34	15	0.14	0.14	0.15	0.06	*	0.07	0.05	0.06	0.04
234-2	16	0.03	0.03	0.04	***	0.10	***	***	***	***
236-4	17	0.25	0.25	0.24	0.04	*	0.05	0.04	0.04	0.02
23-23	18	0.10	0.09	0.09	0.02	*	0.02	0.01	0.02	0.01
235-4	19	0.04	0.04	0.04	0.02	*	0.03	0.02	0.02	0.02
236-25	20	0.17	0.18	0.18	0.04	0.12	0.05	0.03	0.04	0.03
245-4	21	0.20	0.25	0.23	0.09	*	0.13	0.08	0.10	0.06
25-34	22	0.11	0.11	0.13	0.05	0.11	0.06	0.05	0.04	0.02
24-34	23	0.44	0.44	0.44	0.14	*	0.18	0.13	0.13	0.06
235-25	24	0.04	0.05	0.04	0.02	*	0.02	0.02	0.02	0.02
23-34	25	0.24	0.28	0.26	0.05	*	0.06	0.05	0.04	0.02
235-24 and 245-25	26	0.19	0.20	0.20	0.10	*	0.12	0.08	0.09	0.06
234-4	27	0.16	0.16	0.18	0.04	0.12	0.06	0.04	0.04	0.02
245-24	28	0.13	0.14	0.15	0.06	*	0.07	0.05	0.06	0.04
245-23	29	0.11	0.13	0.13	0.04	*	0.04	0.04	0.04	0.02
234-25	30	0.13	0.14	0.15	0.04	*	0.05	0.04	0.04	0.03
234-24	31	0.10	0.10	0.11	0.03	*	0.04	0.03	0.03	0.02
236-34	32	0.24	0.28	0.26	0.08	*	0.10	0.07	0.09	0.06
234-23	33	0.09	0.10	0.10	0.03	*	0.04	0.03	0.03	0.02
34-34	34	0.06	0.07	0.07	0.04	*	0.04	0.03	0.03	0.02
245-34	35	0.19	0.21	0.21	0.14	*	0.15	0.11	0.12	0.08
234-34	36	0.15	0.16	0.16	0.07	*	0.07	0.06	0.06	0.04
Total Mass of Quantified PCB Congeners (mg/Kg)		5.08	5.40	5.32	2.11	NA	2.69	1.86	2.09	1.27

\* analyte not detected in extract

\*\*\* conc. of analyte in extract &lt; 0.05 ng/µl

NA = not applicable

Analyte Concentrations are µg compound / g soil, unless otherwise indicated

File Name:		062N21.TXT	063_SP1.TXT	064N22.TXT	065N23.TXT	066N24.TXT	067N25.TXT	068BLK.TXT	069_SP1.TXT	069BLK.TXT
Sample ID:		N 2-1	PCB Cngr Chk Std 0.1 ng/µl mix #3	N 2-2	N 2-3	N 2-4	N 2-5	ASE Blank #1	PCB Cngr Chk Std 0.1 ng/µl mix #5	ASE Blank #2
Analyte	Libr. #									
biphenyl	1	*	*	*	*	*	*	*	*	*
26-4	2	*	*	*	*	*	*	*	*	*
25-26	3	0.04	*	0.05	0.04	0.06	0.03	*	*	*
24-4	4	0.08	*	0.09	0.08	0.11	0.07	*	*	*
24-26	5	0.02	*	0.02	0.02	0.02	0.02	*	*	*
236-2	6	0.05	*	0.07	0.06	0.08	0.05	*	*	*
23-4	7	0.03	*	0.04	0.04	0.05	0.03	*	*	*
23-26	8	0.02	*	0.03	0.03	0.03	0.02	*	*	*
25-25	9	0.32	*	0.42	0.36	0.46	0.26	*	*	*
24-25	10	0.23	*	0.28	0.25	0.35	0.21	*	0.13	*
24-24	11	0.08	0.13	0.12	0.10	0.13	0.08	*	*	*
23-25	12	0.37	*	0.47	0.40	0.52	0.32	*	*	*
236-3	13	0.02	*	***	0.02	0.03	0.02	*	0.12	*
23-24	14	0.12	0.13	0.14	0.12	0.16	0.10	*	*	*
26-34	15	0.06	*	0.08	0.06	0.09	0.06	*	*	*
234-2	16	0.02	*	0.04	0.02	0.03	0.02	*	*	*
236-4	17	0.20	*	0.27	0.23	0.30	0.19	*	0.14	*
23-23	18	0.06	0.12	0.08	0.07	0.10	0.06	*	*	*
235-4	19	0.03	*	0.04	0.03	0.04	0.02	*	0.14	*
236-25	20	0.15	*	0.19	0.17	0.21	0.14	*	*	*
245-4	21	0.19	*	0.23	0.20	0.26	0.17	*	*	*
25-34	22	0.09	*	0.13	0.10	0.12	0.09	*	*	*
24-34	23	0.36	*	0.44	0.37	0.54	0.32	*	*	*
235-25	24	0.04	0.12	0.04	0.04	0.05	0.03	*	*	*
23-34	25	0.20	*	0.29	0.23	0.31	0.20	*	*	*
235-24 and 245-25	26	0.16	0.12	0.20	0.17	0.24	0.15	*	*	*
234-4	27	0.14	*	0.18	0.14	0.20	0.12	*	*	*
245-24	28	0.12	*	0.14	0.12	0.17	0.10	*	*	*
245-23	29	0.10	*	0.12	0.10	0.14	0.10	*	0.13	*
234-25	30	0.11	*	0.15	0.12	0.16	0.11	*	*	*
234-24	31	0.08	*	0.09	0.09	0.12	0.08	*	0.14	*
236-34	32	0.22	*	0.28	0.22	0.34	0.20	*	*	*
234-23	33	0.07	*	0.09	0.07	0.10	0.07	*	*	*
34-34	34	0.04	*	0.06	0.05	0.07	0.04	*	0.12	*
245-34	35	0.17	0.13	0.20	0.16	0.25	0.15	*	*	*
234-34	36	0.12	0.11	0.16	0.13	0.20	0.12	*	*	*
Total Mass of Quantified PCB Congeners (mg/Kg)		4.11	NA	5.23	4.40	6.06	3.76	NA	NA	NA

\* analyte not detected in extract

\*\*\* conc. of analyte in extract &lt; 0.05 ng/µl

NA = not applicable

Analyte Concentrations are µg compound / g soil, unless otherwise indicated

File Name:		091_1248.TXT	101BLKIS.TXT	102_1.TXT	107_5.TXT	112_10.TXT	116_50.TXT
Sample ID:		Aroclor 1248	Blank Isooctane	Biphenyl Chk Std	Biphenyl Chk Std	Biphenyl Chk Std	Biphenyl Chk Std
Analyte	Libr. #	100 ng/µl	(diluent)	1.0 ng/ml	5.0 ng/ml	10 ng/ml	50 ng/ml
biphenyl	1	***	*	1.15	5.62	9.31	47.3
26-4	2	1.20	*	*	*	*	*
25-26	3	1.07	*	*	*	*	*
24-4	4	5.19	*	*	*	*	*
24-26	5	0.34	*	*	*	*	*
236-2	6	1.09	*	*	*	*	*
23-4	7	1.59	*	*	*	*	*
23-26	8	0.48	*	*	*	*	*
25-25	9	6.52	*	*	*	*	*
24-25	10	4.32	*	*	*	*	*
24-24	11	2.10	*	*	*	*	*
23-25	12	6.13	*	*	*	*	*
236-3	13	0.41	*	*	*	*	*
23-24	14	1.80	*	*	*	*	*
26-34	15	2.11	*	*	*	*	*
234-2	16	0.90	*	*	*	*	*
236-4	17	3.53	*	*	*	*	*
23-23	18	1.10	*	*	*	*	*
235-4	19	0.30	*	*	*	*	*
236-25	20	1.74	*	*	*	*	*
245-4	21	3.83	*	*	*	*	*
25-34	22	7.67	*	*	*	*	*
24-34	23	6.97	*	*	*	*	*
235-25	24	0.29	*	*	*	*	*
23-34	25	3.21	*	*	*	*	*
235-24 and 245-25	26	1.85	*	*	*	*	*
234-4	27	2.71	*	*	*	*	*
245-24	28	1.45	*	*	*	*	*
245-23	29	1.07	*	*	*	*	*
234-25	30	1.20	*	*	*	*	*
234-24	31	0.78	*	*	*	*	*
236-34	32	2.36	*	*	*	*	*
234-23	33	0.63	*	*	*	*	*
34-34	34	0.59	*	*	*	*	*
245-34	35	1.81	*	*	*	*	*
234-34	36	1.48	*	*	*	*	*
Total Mass of Quantified PCB Congeners (mg/Kg)		NA	NA	NA	NA	NA	NA

\* analyte not detected in extract

\*\*\* conc. of analyte in extract &lt; 0.05 ng/µl

NA = not applicable

## PCB Study - Cape Canaveral Pilot Scale

3-Nov-99

13-Dec-99

18-Jan-2000

November 1999			December 1999			March 2000		
SF-1-151			SF-1-160			SF-1-174		
Sample	%H <sub>2</sub> O	pH	Sample	%H <sub>2</sub> O		Sample	%H <sub>2</sub> O	
A1-1	12.5	7.56	A1-1	10.6		A1-1	11.3	
A1-2	15.4	7.55	A1-2	9.9		A1-2	9.1	
A1-3	15.9	7.50	A1-3	10.2		A1-3	8.7	
A1-4	16.0	7.50	A1-4	10.2		A1-4	9.5	
A1-5	N/A	7.36	A1-5	10.0		A1-5	12.4	
A2-1	14.2	7.52	A2-1	16.0		A2-1	11.0	
A2-2	12.9	7.56	A2-2	15.3		A2-2	15.8	
A2-3	N/A	7.37	A2-3	13.8		A2-3	9.4	
A2-4	11.4	7.35	A2-4	13.1		A2-4	12.6	
A2-5	N/A	7.45	A2-5	14.4		A2-5	12.6	
N1-1	N/A	6.92	N1-1	6.4		N1-1	4.9	
N1-2	N/A	6.91	N1-2	8.9		N1-2	5.1	
N1-3	15.6	6.81	N1-3	N/A		N1-3	4.7	
N1-4	16.3	7.30	N1-4	7.6		N1-4	7.2	
N1-5	16.9	6.78	N1-5	5.5		N2-1	5.9	
N2-1	13.1	6.56	N2-1	8.6		N2-2	6.0	
N2-2	14.6	6.81	N2-2	10.1		N2-3	8.2	
N2-3	N/A	6.90	N2-3	8.6		N2-4	8.4	
N2-4	14.1	7.37	N2-4	10.9		X1-1	13.1	
N2-5	14.4	6.76	N2-5	8.7		X1-2	10.7	
X1-1	20.5	7.17	X1-1	15.6		X1-3	11.3	
X1-2	24.1	6.93	X1-2	13.2		X2-4	N/A	
X1-3	22.1	6.88	X1-3	15.3		X1-5	N/A	
X1-4	20.2	6.78	X1-4	11.3		N1-5	N/A	
X1-5	22.1	6.70	X1-5	12.2		N2-5	N/A	
X2-1	N/A	6.76	X2-1	14.7		X2-1	12.8	
X2-2	19.8	6.84	X2-2	14.4		X2-2	13.5	
X2-3	22.1	5.88	X2-3	14.5		X2-3	12.2	
X2-4	N/A	6.75	X2-4	14.9		X2-4	N/A	
X2-5	23.1	6.91	X2-5	15.9		X2-5	N/A	

No pH values for Dec and March samples

Results from Seventh Sampling Event  
(Day 516)

Sampling Date: 13-December-1999

R.S. Kerr Laboratory Service Request Number: SF-0-160

**\* PCB and pH results from the seventh sampling event were never received  
from the laboratory and are therefore not included**

## PCB Study - Cape Canaveral Pilot Scale

3-Nov-99

13-Dec-99

18-Jan-2000

November 1999			December 1999			March 2000		
SF-1-151			SF-1-160			SF-1-174		
Sample	%H <sub>2</sub> O	pH	Sample	%H <sub>2</sub> O		Sample	%H <sub>2</sub> O	
A1-1	12.5	7.56	A1-1	10.6		A1-1	11.3	
A1-2	15.4	7.55	A1-2	9.9		A1-2	9.1	
A1-3	15.9	7.50	A1-3	20.2		A1-3	8.7	
A1-4	16.0	7.50	A1-4	10.2		A1-4	9.5	
A1-5	N/A	7.36	A1-5	10.0		A1-5	12.4	
A2-1	14.2	7.52	A2-1	16.0		A2-1	11.0	
A2-2	12.9	7.56	A2-2	15.3		A2-2	15.8	
A2-3	N/A	7.37	A2-3	13.8		A2-3	9.4	
A2-4	11.4	7.35	A2-4	13.1		A2-4	12.6	
A2-5	N/A	7.45	A2-5	14.4		A2-5	12.6	
N1-1	N/A	6.92	N1-1	6.4		N1-1	4.9	
N1-2	N/A	6.91	N1-2	8.9		N1-2	5.1	
N1-3	15.6	6.81	N1-3	N/A		N1-3	4.7	
N1-4	16.3	7.30	N1-4	7.6		N1-4	7.2	
N1-5	16.9	6.78	N1-5	5.5		N2-1	5.9	
N2-1	13.1	6.56	N2-1	8.6		N2-2	6.0	
N2-2	14.6	6.81	N2-2	10.1		N2-3	8.2	
N2-3	N/A	6.90	N2-3	8.6		N2-4	8.4	
N2-4	14.1	7.37	N2-4	10.9		X1-1	13.1	
N2-5	14.4	6.76	N2-5	8.7		X1-2	10.7	
X1-1	20.5	7.17	X1-1	15.6		X1-3	11.3	
X1-2	24.1	6.93	X1-2	13.2		X2-4	N/A	
X1-3	22.1	6.88	X1-3	15.3		X1-5	N/A	
X1-4	20.2	6.78	X1-4	11.3		N2-5	N/A	
X1-5	22.1	6.70	X1-5	12.2		N2-5	N/A	
X2-1	N/A	6.76	X2-1	14.7		X2-1	12.8	
X2-2	19.8	6.84	X2-2	14.4		X2-2	13.5	
X2-3	22.1	5.88	X2-3	14.5		X2-3	12.2	
X2-4	N/A	6.75	X2-4	14.9		X2-4	N/A	
X2-5	23.1	6.91	X2-5	15.9		X2-5	N/A	

No pH values for Dec and March samples

Results from Eighth Sampling Event  
(Day 552)

Sampling Date: 18-January-2000

R.S. Kerr Laboratory Service Request Number: SF-0-174

Analyte Concentrations are µg compound / g soil, unless otherwise indicated

File Name:		001BLK08.TXT	002_1PS.TXT	003A11.TXT	004A12.TXT	005A13.TXT	006A14.TXT	007A15A.TXT	008_11.TXT	009A15B.TXT
Sample ID:		Blank Isooctane	PCB Cngr Chk Std	A 1-1	A 1-2	A 1-3	A 1-4	A 1-5 (A)	PCB Cngr Chk Std	A 1-5 (B)
Analyte	Libr. #	(diluent)	0.5 ng/µl mix #1						1.0 ng/µl mix #1	
biphenyl	1	*	***	0.03	***	***	***	***	***	***
26-4	2	*	*	0.04	0.04	0.02	0.02	0.06	*	0.06
25-26	3	*	*	0.16	0.15	0.10	0.11	0.16	*	0.16
24-4	4	*	0.61	0.06	0.06	0.04	0.04	0.06	1.18	0.07
24-26	5	*	*	0.05	0.05	0.04	0.04	0.06	*	0.05
236-2	6	*	*	0.21	0.18	0.13	0.17	0.19	*	0.20
23-4	7	*	0.58	0.07	0.05	0.04	0.04	0.07	1.21	0.06
23-26	8	*	*	0.09	0.07	0.05	0.07	0.08	*	0.09
25-25	9	*	0.68	1.28	1.12	0.79	0.87	1.07	1.19	1.20
24-25	10	*	*	0.92	0.84	0.59	0.71	0.84	*	0.94
24-24	11	*	*	0.30	0.30	0.20	0.24	0.29	*	0.32
23-25	12	*	0.65	1.55	1.34	0.97	1.06	1.28	1.23	1.33
236-3	13	*	*	0.08	0.08	0.06	0.05	0.06	*	0.07
23-24	14	*	*	0.40	0.35	0.27	0.31	0.32	*	0.39
26-34	15	*	0.59	0.27	0.24	0.16	0.18	0.22	1.31	0.24
234-2	16	*	*	0.09	0.08	0.05	0.06	0.05	*	0.08
236-4	17	*	*	0.76	0.68	0.46	0.54	0.60	*	0.71
23-23	18	*	*	0.25	0.23	0.16	0.18	0.21	*	0.25
235-4	19	*	*	0.07	0.06	0.04	0.06	0.06	*	0.06
236-25	20	*	*	0.52	0.45	0.32	0.39	0.43	*	0.45
245-4	21	*	0.64	0.53	0.54	0.38	0.45	0.51	1.30	0.61
25-34	22	*	*	0.08	0.06	0.05	0.05	0.07	*	0.07
24-34	23	*	0.61	1.15	1.18	0.78	0.93	1.02	1.17	1.13
235-25	24	*	*	0.09	0.09	0.06	0.07	0.09	*	0.07
23-34	25	*	0.62	0.68	0.61	0.46	0.57	0.58	1.32	0.70
235-24 and 245-25	26	*	*	0.50	0.48	0.33	0.37	0.44	*	0.47
234-4	27	*	*	0.46	0.47	0.30	0.33	0.38	*	0.48
245-24	28	*	0.67	0.35	0.34	0.22	0.26	0.32	1.26	0.35
245-23	29	*	*	0.30	0.28	0.19	0.23	0.25	*	0.31
234-25	30	*	0.59	0.32	0.32	0.22	0.26	0.28	1.30	0.31
234-24	31	*	*	0.23	0.19	0.15	0.15	0.17	*	0.20
236-34	32	*	0.63	0.66	0.63	0.44	0.52	0.58	1.28	0.61
234-23	33	*	0.65	0.20	0.18	0.14	0.16	0.17	1.31	0.20
34-34	34	*	*	0.11	0.11	0.07	0.09	0.11	*	0.11
245-34	35	*	*	0.45	0.45	0.29	0.35	0.42	*	0.46
234-34	36	*	*	0.37	0.32	0.24	0.27	0.34	*	0.36
Total Mass of Quantified PCB Congeners (mg/Kg)		NA	NA	13.66	12.63	8.80	10.19	11.84	NA	13.21

\* analyte not detected in extract

\*\*\* conc. of analyte in extract &lt; 0.05 ng/µl

NA = not applicable

Analyte Concentrations are µg compound / g soil, unless otherwise indicated

File Name:		010A21.TXT	011A22.TXT	012A23.TXT	013A24.TXT	014_2PS.TXT	015A25.TXT	016X11A.TXT	017X11B.TXT	018X12.TXT
Sample ID:		A 2-1	A 2-2	A 2-3	A 2-4	PCB Cngr Chk Std 0.5 ng/µl mix #2	A 2-5	X 1-1 (A)	X 1-1 (B)	X 1-2
Analyte	Libr. #									
biphenyl	1	0.13	8.98	0.04	0.07	*	1.34	***	***	*
26-4	2	0.14	0.21	0.09	0.24	0.57	0.31	***	0.03	0.02
25-26	3	0.29	0.45	0.23	0.43	*	0.57	0.05	0.07	0.05
24-4	4	0.10	0.11	0.06	0.11	*	0.19	0.07	0.12	0.08
24-26	5	0.09	0.13	0.06	0.12	*	0.16	0.02	0.03	0.02
236-2	6	0.35	0.52	0.27	0.45	0.52	0.57	0.06	0.08	0.06
23-4	7	0.11	0.19	0.04	0.12	*	0.26	0.04	0.06	0.03
23-26	8	0.16	0.22	0.12	0.17	0.50	0.24	0.03	0.04	0.03
25-25	9	1.99	2.86	1.45	2.56	*	3.41	0.30	0.40	0.29
24-25	10	1.47	2.30	1.14	1.92	*	2.49	0.22	0.32	0.23
24-24	11	0.52	0.77	0.42	0.67	*	0.94	0.09	0.11	0.09
23-25	12	2.32	3.16	1.79	2.89	*	3.87	0.36	0.46	0.35
236-3	13	0.14	0.19	0.08	0.18	*	0.25	***	0.03	0.02
23-24	14	0.63	0.98	0.51	0.83	*	1.09	0.11	0.14	0.10
26-34	15	0.56	0.86	0.45	0.76	*	1.11	0.10	0.14	0.11
234-2	16	0.16	0.20	0.11	0.19	0.55	0.28	0.03	0.04	0.03
236-4	17	1.27	1.74	0.96	1.51	*	2.06	0.22	0.26	0.19
23-23	18	0.39	0.58	0.31	0.49	*	0.65	0.07	0.10	0.07
235-4	19	0.10	0.15	0.08	0.14	*	0.19	0.03	0.04	0.03
236-25	20	0.75	1.16	0.64	1.04	0.60	1.35	0.13	0.19	0.13
245-4	21	0.95	1.38	0.78	1.27	*	1.80	0.17	0.24	0.18
25-34	22	0.14	0.17	0.10	0.17	0.61	0.29	0.09	0.12	0.08
24-34	23	1.88	2.93	1.47	2.63	*	3.56	0.36	0.42	0.34
235-25	24	0.12	0.18	0.11	0.17	*	0.28	0.03	0.04	0.03
23-34	25	1.18	1.65	0.83	1.48	*	2.05	0.21	0.27	0.20
235-24 and 245-25	26	0.84	1.25	0.62	1.13	*	1.41	0.17	0.19	0.15
234-4	27	0.78	1.12	0.58	1.06	0.62	1.28	0.13	0.18	0.13
245-24	28	0.57	0.84	0.40	0.74	*	0.99	0.11	0.13	0.11
245-23	29	0.51	0.66	0.37	0.65	*	0.83	0.10	0.12	0.10
234-25	30	0.55	0.82	0.40	0.74	*	0.93	0.11	0.13	0.11
234-24	31	0.33	0.48	0.24	0.38	*	0.60	0.08	0.08	0.07
236-34	32	1.08	1.44	0.85	1.37	*	1.75	0.22	0.27	0.21
234-23	33	0.30	0.45	0.25	0.43	*	0.52	0.07	0.09	0.07
34-34	34	0.18	0.24	0.13	0.24	*	0.29	0.05	0.06	0.05
245-34	35	0.74	0.98	0.57	1.01	*	1.19	0.15	0.19	0.15
234-34	36	0.59	0.84	0.45	0.76	*	0.96	0.12	0.15	0.13
Total Mass of Quantified PCB Congeners (mg/Kg)		22.27	32.20	16.97	29.04	NA	38.75	4.12	5.34	4.04

\* analyte not detected in extract

\*\*\* conc. of analyte in extract &lt; 0.05 ng/µl

biphenyl conc. for "A 2-2" is an ESTIMATE ONLY

NA = not applicable

Analyte Concentrations are µg compound / g soil, unless otherwise indicated

File Name:		019X13.TXT	020_21.TXT	021X21.TXT	022X22.TXT	023X23A.TXT	024X23B.TXT	025N11.TXT	026_3P5.TXT	027A13D.TXT
Sample ID:		X 1-3	PCB Cngr Chk Std 1.0 ng/µl mix #2	X 2-1	X 2-2	X 2-3 (A)	X 2-3 (B)	N 1-1	PCB Cngr Chk Std 0.5 ng/µl mix #3	A 1-3 lab dup
Analyte	Libr. #									
biphenyl	1	***	*	***	*	***	***	*	*	***
26-4	2	***	1.13	0.03	0.02	***	0.02	0.07	*	0.02
25-26	3	0.05	*	0.06	0.06	0.05	0.06	0.05	*	0.10
24-4	4	0.08	*	0.10	0.10	0.08	0.08	0.13	*	0.04
24-26	5	0.03	*	0.03	0.03	0.03	0.03	0.02	*	0.03
236-2	6	0.06	1.10	0.07	0.07	0.07	0.07	***	*	0.15
23-4	7	0.04	*	0.05	0.04	0.04	0.05	***	*	0.04
23-26	8	0.03	1.03	0.03	0.03	0.03	0.03	***	*	0.05
25-25	9	0.30	*	0.36	0.41	0.35	0.37	0.06	*	0.79
24-25	10	0.22	*	0.30	0.31	0.25	0.26	0.04	*	0.62
24-24	11	0.09	*	0.12	0.11	0.09	0.09	0.07	0.63	0.20
23-25	12	0.37	*	0.49	0.48	0.39	0.39	0.03	*	0.97
236-3	13	0.02	*	0.03	0.03	0.02	0.02	***	*	0.05
23-24	14	0.11	*	0.14	0.14	0.12	0.12	0.02	0.62	0.28
26-34	15	0.12	*	0.13	0.14	0.11	0.13	0.03	*	0.17
234-2	16	0.03	1.10	0.04	0.03	0.03	0.04	***	*	0.05
236-4	17	0.20	*	0.25	0.24	0.20	0.24	0.02	*	0.51
23-23	18	0.08	*	0.10	0.09	0.07	0.08	0.01	0.57	0.16
235-4	19	0.03	*	0.03	0.03	0.03	0.04	0.01	*	0.05
236-25	20	0.15	1.40	0.19	0.18	0.15	0.16	0.02	*	0.38
245-4	21	0.19	*	0.22	0.23	0.18	0.22	0.06	*	0.41
25-34	22	0.10	1.35	0.12	0.11	0.10	0.11	0.02	*	0.05
24-34	23	0.39	*	0.45	0.47	0.40	0.42	0.08	*	0.81
235-25	24	0.04	*	0.05	0.05	0.04	0.03	0.01	0.60	0.07
23-34	25	0.22	*	0.27	0.27	0.21	0.24	0.03	*	0.48
235-24 and 245-25	26	0.16	*	0.20	0.20	0.18	0.18	0.05	0.63	0.36
234-4	27	0.14	1.28	0.17	0.18	0.13	0.16	0.03	*	0.32
245-24	28	0.12	*	0.15	0.15	0.12	0.14	0.03	*	0.24
245-23	29	0.11	*	0.13	0.12	0.10	0.11	0.02	*	0.20
234-25	30	0.12	*	0.14	0.15	0.12	0.13	0.03	*	0.25
234-24	31	0.10	*	0.11	0.08	0.08	0.08	0.02	*	0.14
236-34	32	0.23	*	0.25	0.27	0.23	0.24	0.05	*	0.50
234-23	33	0.08	*	0.09	0.10	0.08	0.08	0.02	*	0.14
34-34	34	0.05	*	0.06	0.07	0.05	0.06	0.02	*	0.08
245-34	35	0.18	*	0.19	0.20	0.17	0.17	0.08	0.61	0.34
234-34	36	0.13	*	0.15	0.16	0.14	0.14	0.04	0.63	0.27
Total Mass of Quantified PCB Congeners (mg/Kg)		4.38	NA	5.28	5.38	4.46	4.78	1.17	NA	9.32

\* analyte not detected in extract

\*\*\* conc. of analyte in extract &lt; 0.05 ng/µl

NA = not applicable

Analyte Concentrations are µg compound / g soil, unless otherwise indicated

File Name:		028N12.TXT	029N13.TXT	030N14.TXT	031N21.TXT	032_31.TXT	033N22A.TXT	034N22B.TXT	035N23.TXT	036N24.TXT
Sample ID:		N 1-2	N 1-3	N 1-4	N 2-1	PCB Cngr Chk Std 1.0 ng/µl mix #3	N 2-2 (A)	N 2-2 (B)	N 2-3	N 2-4
Analyte	Libr. #									
biphenyl	1	*	*	*	*	*	*	*	*	*
26-4	2	0.10	0.13	0.16	***	*	***	***	***	***
25-26	3	0.07	0.14	0.13	0.02	*	0.03	0.03	0.03	0.04
24-4	4	0.18	0.22	0.33	0.05	*	0.05	0.05	0.06	0.07
24-26	5	0.03	0.05	0.05	***	*	***	***	***	***
236-2	6	0.01	0.07	0.04	0.04	*	0.04	0.05	0.05	0.05
23-4	7	0.01	0.06	0.03	0.02	*	0.02	0.03	0.03	0.03
23-26	8	***	0.03	0.02	0.01	*	0.01	0.02	0.02	0.02
25-25	9	0.08	0.58	0.33	0.23	*	0.22	0.27	0.29	0.31
24-25	10	0.06	0.43	0.19	0.18	*	0.18	0.22	0.22	0.25
24-24	11	0.10	0.23	0.18	0.06	1.14	0.06	0.07	0.08	0.08
23-25	12	0.04	0.55	0.22	0.27	*	0.27	0.31	0.35	0.39
236-3	13	***	0.05	0.02	0.02	*	0.01	0.02	0.02	0.02
23-24	14	0.02	0.16	0.07	0.07	1.16	0.08	0.09	0.10	0.11
26-34	15	0.05	0.22	0.12	0.04	*	0.04	0.05	0.05	0.06
234-2	16	***	0.06	0.02	0.02	*	0.02	0.02	0.03	0.03
236-4	17	0.03	0.33	0.14	0.16	*	0.14	0.19	0.18	0.20
23-23	18	0.01	0.11	0.06	0.05	1.05	0.05	0.06	0.06	0.07
235-4	19	0.02	0.04	0.03	0.01	*	0.01	0.02	0.02	0.02
236-25	20	0.03	0.26	0.12	0.11	*	0.11	0.16	0.14	0.16
245-4	21	0.08	0.39	0.21	0.14	*	0.15	0.18	0.18	0.20
25-34	22	0.03	0.12	0.07	0.07	*	0.07	0.08	0.08	0.10
24-34	23	0.11	0.74	0.35	0.29	*	0.31	0.36	0.34	0.41
235-25	24	0.02	0.06	0.04	0.02	1.12	0.02	0.03	0.03	0.03
23-34	25	0.03	0.38	0.15	0.16	*	0.16	0.20	0.20	0.23
235-24 and 245-25	26	0.08	0.37	0.19	0.12	1.19	0.13	0.16	0.15	0.18
234-4	27	0.03	0.27	0.12	0.11	*	0.11	0.14	0.13	0.15
245-24	28	0.05	0.24	0.12	0.09	*	0.09	0.10	0.11	0.12
245-23	29	0.03	0.19	0.08	0.07	*	0.07	0.09	0.10	0.11
234-25	30	0.03	0.21	0.09	0.09	*	0.08	0.11	0.11	0.12
234-24	31	0.02	0.12	0.07	0.05	*	0.06	0.07	0.07	0.08
236-34	32	0.07	0.43	0.19	0.17	*	0.17	0.22	0.23	0.23
234-23	33	0.02	0.11	0.06	0.05	*	0.05	0.06	0.06	0.07
34-34	34	0.03	0.08	0.05	0.03	*	0.03	0.04	0.03	0.04
245-34	35	0.12	0.37	0.21	0.13	1.17	0.12	0.16	0.16	0.16
234-34	36	0.05	0.25	0.12	0.10	1.32	0.09	0.12	0.11	0.14
Total Mass of Quantified PCB Congeners (mg/Kg)		1.63	8.07	4.36	3.05	NA	3.06	3.75	3.78	4.26

\* analyte not detected in extract

\*\*\* conc. of analyte in extract &lt; 0.05 ng/µl

NA = not applicable

Analyte Concentrations are µg compound / g soil, unless otherwise indicated

File Name:		0371260.TXT	038_5P5.TXT	039BLK.TXT	040BLK.TXT	041_51.TXT	042_1260.TXT	043BLKIS.TXT	044_P5.TXT	045_1.TXT
Sample ID:		ASE Soil Extract	PCB Cngr Chk Std	ASE Blank #1	ASE Blank #2	PCB Cngr Chk Std	Aroclor 1260	Blank Isooctane	biphenyl	biphenyl
Analyte	Libr. #	Aroclor 1260	0.5 ng/µl mix #5			1.0 ng/µl mix #5	5.0 ng/µl	(diluent)	0.5 ng/µl	1.0 ng/µl
biphenyl	1	*	*	*	*	*	*	*	0.47	0.92
26-4	2	*	*	*	*	*	*	*	*	*
25-26	3	*	*	*	*	*	*	*	*	*
24-4	4	*	*	*	*	*	*	*	*	*
24-26	5	*	*	*	*	*	*	*	*	*
236-2	6	*	*	*	*	*	*	*	*	*
23-4	7	*	*	*	*	*	*	*	*	*
23-26	8	*	*	*	*	*	*	*	*	*
25-25	9	***	*	*	*	*	***	*	*	*
24-25	10	*	0.62	*	*	1.31	*	*	*	*
24-24	11	*	*	*	*	*	*	*	*	*
23-25	12	*	*	*	*	*	*	*	*	*
236-3	13	*	0.61	*	*	1.35	*	*	*	*
23-24	14	*	*	*	*	*	*	*	*	*
26-34	15	*	*	*	*	*	*	*	*	*
234-2	16	*	*	*	*	*	*	*	*	*
236-4	17	*	0.59	*	*	1.37	*	*	*	*
23-23	18	*	*	*	*	*	*	*	*	*
235-4	19	*	0.57	*	*	1.28	*	*	*	*
236-25	20	2.55	*	*	*	*	0.17	*	*	*
245-4	21	*	*	*	*	*	*	*	*	*
25-34	22	*	*	*	*	*	*	*	*	*
24-34	23	*	*	*	*	*	*	*	*	*
235-25	24	*	*	*	*	*	*	*	*	*
23-34	25	*	*	*	*	*	*	*	*	*
235-24 and 245-25	26	2.55	*	*	*	*	0.18	*	*	*
234-4	27	*	*	*	*	*	*	*	*	*
245-24	28	*	*	*	*	*	*	*	*	*
245-23	29	*	0.60	*	*	1.31	*	*	*	*
234-25	30	***	*	*	*	*	***	*	*	*
234-24	31	*	0.59	*	*	1.38	*	*	*	*
236-34	32	1.18	*	*	*	*	0.08	*	*	*
234-23	33	***	*	*	*	*	*	*	*	*
34-34	34	***	0.57	*	*	1.16	***	*	*	*
245-34	35	***	*	*	*	*	***	*	*	*
234-34	36	***	*	*	*	*	***	*	*	*
Total Mass of Quantified PCB Congeners (mg/Kg)		6.28	NA	NA	NA	NA	NA	NA	NA	NA

\* analyte not detected in extract

\*\*\* conc. of analyte in extract &lt; 0.05 ng/µl

NA = not applicable

Analyte Concentrations are µg compound / g soil, unless otherwise indicated

File Name:		046BLKIS.TXT							
Sample ID:		Blank Isooctane							
Analyte	Libr. #	100 ng/ml							
biphenyl	1	*							
26-4	2	*							
25-26	3	*							
24-4	4	*							
24-26	5	*							
236-2	6	*							
23-4	7	*							
23-26	8	*							
25-25	9	*							
24-25	10	*							
24-24	11	*							
23-25	12	*							
236-3	13	*							
23-24	14	*							
26-34	15	*							
234-2	16	*							
236-4	17	*							
23-23	18	*							
235-4	19	*							
236-25	20	*							
245-4	21	*							
25-34	22	*							
24-34	23	*							
235-25	24	*							
23-34	25	*							
235-24 and 245-25	26	*							
234-4	27	*							
245-24	28	*							
245-23	29	*							
234-25	30	*							
234-24	31	*							
236-34	32	*							
234-23	33	*							
34-34	34	*							
245-34	35	*							
234-34	36	*							
Total Mass of Quantified PCB Congeners (mg/Kg)		NA							

\* analyte not detected in extract

\*\*\* conc. of analyte in extract &lt; 0.05 ng/µl

NA = not applicable

## PCB Study - Cape Canaveral Pilot Scale

3-Nov-99

13-Dec-99

18-Jan-2000

November 1999			December 1999			March 2000		
SF-1-151			SF-1-160			SF-1-174		
Sample	%H <sub>2</sub> O	pH	Sample	%H <sub>2</sub> O		Sample	%H <sub>2</sub> O	
A1-1	12.5	7.56	A1-1	10.6		A1-1	11.3	
A1-2	15.4	7.55	A1-2	9.9		A1-2	9.1	
A1-3	15.9	7.50	A1-3	10.2		A1-3	8.7	
A1-4	16.0	7.50	A1-4	10.2		A1-4	9.5	
A1-5	N/A	7.36	A1-5	10.0		A1-5	12.4	
A2-1	14.2	7.52	A2-1	16.0		A2-1	11.0	
A2-2	12.9	7.56	A2-2	15.3		A2-2	15.8	
A2-3	N/A	7.37	A2-3	13.8		A2-3	9.4	
A2-4	11.4	7.35	A2-4	13.1		A2-4	12.6	
A2-5	N/A	7.45	A2-5	14.4		A2-5	12.6	
N1-1	N/A	6.92	N1-1	6.4		N1-1	4.9	
N1-2	N/A	6.91	N1-2	8.9		N1-2	5.1	
N1-3	15.6	6.81	N1-3	N/A		N1-3	4.7	
N1-4	16.3	7.30	N1-4	7.6		N1-4	7.2	
N1-5	16.9	6.78	N1-5	5.5		N2-1	5.9	
N2-1	13.1	6.56	N2-1	8.6		N2-2	6.0	
N2-2	14.6	6.81	N2-2	10.1		N2-3	8.2	
N2-3	N/A	6.90	N2-3	8.6		N2-4	8.4	
N2-4	14.1	7.37	N2-4	10.9		X1-1	13.1	
N2-5	14.4	6.76	N2-5	8.7		X1-2	10.7	
X1-1	20.5	7.17	X1-1	15.6		X1-3	11.3	
X1-2	24.1	6.93	X1-2	13.2		X2-4	N/A	
X1-3	22.1	6.88	X1-3	15.3		X1-5	N/A	
X1-4	20.2	6.78	X1-4	11.3		N1-5	N/A	
X1-5	22.1	6.70	X1-5	12.2		N2-5	N/A	
X2-1	N/A	6.76	X2-1	14.7		X2-1	12.8	
X2-2	19.8	6.84	X2-2	14.4		X2-2	13.5	
X2-3	22.1	5.88	X2-3	14.5		X2-3	12.2	
X2-4	N/A	6.75	X2-4	14.9		X2-4	N/A	
X2-5	23.1	6.91	X2-5	15.9		X2-5	N/A	

No pH values for Dec and March samples